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THE FLAVOR STABILITY OF CANNED BUTTER

by

Harold A. Hansen

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## ABSTRACT

This investigation was undertaken in an endeavour to develop a butter with maximum flavor stability at storage temperatures as high as 90°F.

Canned butter did not have sufficient flavor stability when held at room temperature or 95°F. to warrant storage at these temperatures. To increase the keeping quality, butters were subjected to various changes in processing and composition. Flavor stability of the modified butters was determined by organoleptic evaluation which was supplemented by estimations of microorganisms and by determinations of free fat acidity and fat oxidation.

Heat treatment of butter in sealed cans at 170° to 180°F. for 10 minutes followed by re-emulsification of the serum and butterfat generally prevented the development of bacterially induced off flavors at room temperature storage, in addition to checking growth of the surviving bacteria at that temperature. Growth of the surviving bacteria in the modified butter stored at 90°F. was controlled by prevention of oiling off and serum separation. In the absence of bacterially induced off flavors in the modified butter, a tallowy flavor caused by butterfat oxidation developed. Incorporation of antioxidants into these butters did not retard the development of tallowiness.



Nitrogen packing, subsequent to almost complete removal of the oxygen from the canned modified butters, was found to inhibit butterfat oxidation sufficiently to prevent the onset of a tallowy flavor. These butters have been stored for 19 months at 70°F. without serious flavor deterioration. Similar butters stored at 90°F. developed a cheesy and eventually a rancid flavor.

A nitrogen packed butter concentrate, similar to the Australian product, in which the moisture content was reduced to 0.2 to 0.3%, has been stored for as long as 12 months at 90°F. without developing a tallowy, rancid or cheesy flavor. The data available to date in this investigation indicate that butter concentrate may be a more suitable product than the nitrogen packed modified butter especially for use at storage temperatures as high as 90°F.



THE UNIVERSITY OF ALBERTA

THE FLAVOR STABILITY OF CANNED BUTTER

A DISSERTATION

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## THE FLAVOR STABILITY OF CANNED BUTTER

### INTRODUCTION

Canned butter has been used in Canada over a considerable period of time by prospectors and by others stationed in the northern areas. Hermetically sealed cans have provided the best package available to meet the severe transportation and storage requirements of this region. Little attention, however, has been given to the flavor stability of the product.

Prior to World War II there was almost no interest in the keeping quality of butter at temperatures above those used in long term storage (0°F.) and, consequently, little research was carried out in this field. Following the war, the few published results indicated that the flavor stability of butter is unsatisfactory at storage temperatures above those employed in cold storage. These results are not surprising in view of the fact that butter is not a sterile product. Under suitable conditions, such as incubation at summer temperatures, the microorganisms, capable of causing off flavors, will grow. Pont (1945) found that Australian canned butter showed a pronounced tendency to deteriorate in flavor when stored in a hot climate. Remaley (1948), in summarizing the experiences of the Quartermaster



Food and Container Institute of the United States armed forces in the development of spreads for bread throughout World War II, reported that a product with the necessary flavor stability was not developed from either butter or vegetable oils.

At the present time, with a continued interest on the part of the armed services and with increasing activity in Northern Canada, there would appear to be a need for a canned butter which would have improved keeping quality at storage and transportation temperatures as high as 90°F. This investigation was undertaken in an endeavour to develop a butter with the maximum protection against flavor deterioration.

#### HISTORICAL

#### Keeping Quality of Butter

While first grade butter kept in low temperature storage for several months may lose nothing more than some of its original bloom, Barnicoat (1946) found that even a low temperature level of storage (14°F.) does not completely retard the appearance of flavor defects. The predominating off flavor was staleness which Barnicoat and Palmer (1939)



called an incipient oxidation defect. Wiley (1939) reported that oxidation of the fat is one of the most serious causes of deterioration in butter submitted to long periods in cold storage.

Hammer and Long (1941) found that bacterial growth capable of causing flavor defects is largely prevented by storage at temperatures of 0°C. (32°F.) or below. At temperatures between 14° and 60°F., Barnicoat reported the average rate of deterioration of first grade butter to be related directly to time and temperature.

Grant et al. (1948) observed that no appreciable oxidative decomposition could be detected in the butterfat from butters stored at 30°, 50°, 70° and 90°F. However, they found that stability of the serum was the limiting factor in keeping quality. White (1946) studied nine creamery and two laboratory churned butters packed in 2 and 3 oz. tinned cans which were stored at 10°, 40°, 70° and 85°F. for varying periods of time. The principal flavor defects at the higher temperatures were rancid and cheesy which appeared after four to eight weeks of storage.

According to Pont (1945), tinned butter showed pronounced liability to deterioration when used in hot climates. The principal defects were rancid, cheesy and putrid flavors caused by bacterial activity. When butter was subjected to



temperatures above its melting point, deterioration was severe and rapid. Wiley and Coombs (1946) claimed that at atmospheric temperatures when rancidity did not show up on butter oxidative flavors did.

Butter packaged in cans appeared to have a better keeping quality than butter packaged in parchment. Grant et al. concluded that butter in cans deteriorated less rapidly than print samples although differences were less pronounced at 70° and 90°F. than at 30° and 50°F. Canned samples did not change appreciably in quality during 45 weeks' storage at 30°F. Barnicoat (1938) found that butter packed in tins was preferable to butter kept in 56-pound wooden boxes after storage of from three to six months at 14°F. The tin containers gave complete protection against outside taints.

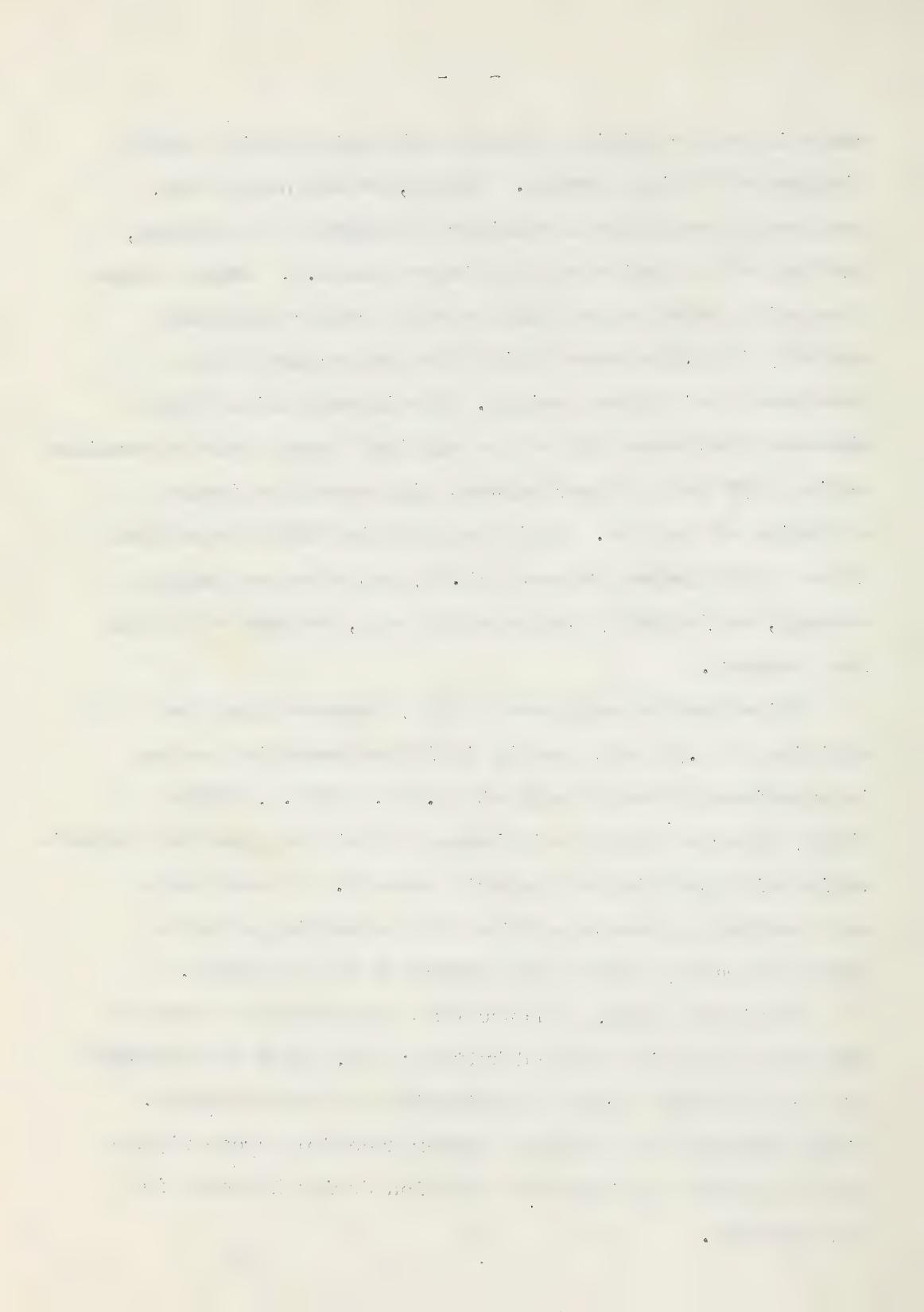
Wiley reported that acidity, starter organisms, salt and low pasteurization temperatures each favor the development of oxidized flavors in butter. Barnicoat (1951) gave the principal factors influencing the storage life of New Zealand butter as: quality of cream received, acidity of cream at churning (or pH of the butter when made), salt content, degree of contamination by copper or iron, temperature level and duration of storage. He concluded that metallic contamination is the most important factor concerned with deterioration. Barnicoat (1950) found that iron tended to



cause in butter initial objectionable taints which usually disappeared during storage. Copper, on the other hand, promoted by catalysis a considerable degree of oxidation, particularly in butters of pH lower than 6.5. Butters made from acid creams containing traces of copper decreased markedly in grade score during storage because of the development of tallowy taints. Martin and Julien (1951) observed that about 25% of the oxidized Quebec butters examined during 1950 had a copper content high enough to induce oxidation of the fat. Julien and Martin (1952) found that 16 out of 68 Quebec butters (23.5%) deteriorated during storage, as judged by the peroxide test, because of a high iron content.

While Hoecker and Hammer (1945) reported that the addition of 1.5% salt greatly inhibited bacterial action in experimental butter held at 15.6°C. (60°F.), Slater (1946) observed that the influence of salt on growth of bacteria varies with the kind of bacteria present. In some cases salt entirely prevents a defect from appearing while in others it merely delays the appearance of the defect.

Barnicoat (1948, a) noted that no oxidation of the fat had taken place in unsalted butters; but, there was evidence of a considerable degree of oxidation in salted samples. Wiley reported that unsalted butter made from cream ripened to pH 5 showed considerable oxidation during 18 weeks in cold storage.



At various storage temperatures Holm et al. (1938) noted that in butters made from creams of increasing acidity the rate of oxidation increased.

#### Flavor Defects and Their Cause

##### (a) Hydrolytic Rancidity

Shipstead and Tarassuk (1953) found that the flavor of hydrolytic rancidity is characterized by a bitter taste and a sharp unpleasant odor resembling butyric acid and it results from the partial hydrolysis of the fat with the resulting appearance of free fatty acids from butyric to myristic.

Lea (1939) concluded that the lipolytic enzymes of numerous species of bacteria and molds, both aerobic and anaerobic, are capable of hydrolyzing fats.

Hammer (1948) reported that in the early studies of Orla-Jensen, a number of species of bacteria were found which produced rancidity, including Pseudomonas fluorescens and Serratia marcescens. Collins and Hammer (1934) isolated 159 lipolytic bacterial cultures from sources such as water, air, dairy plant equipment and dairy products. Common species found were P. fragi, Achromobacter lipolyticum and P. fluorescens.

Hammer and Long (1941) observed that in butter a number of factors apparently vary in their effect on different



organisms and a factor which largely prevents the growth of one species may have little influence on another. Mainly, the growth of bacteria in butter is influenced by the physical condition, salt content, temperature of storage, acidity and air supply.

Molds are also active in hydrolyzing fat, one of the most common being Oospora lactis. Macy and Gibson (1937) studied 61 cultures of this mold isolated from butter, finding that all of them hydrolyzed cottonseed oil and butterfat and also liquefied gelatin.

Today, all creamery butter is made from pasteurized cream. Hammer noted that proper pasteurization of cream is recognized as one of the important factors in manufacturing butter that will keep satisfactorily during distribution and consumption. When butter made from pasteurized cream with or without salt shows bacterial deterioration, the causitive organisms are normally present in the product because of inefficient pasteurization or because of contamination subsequent to heating.

Heat treatment of the cream is a possible way of eliminating the effect of the natural lipolytic enzyme. Rogers (1904) found that lipase was entirely destroyed at 60°C. (140°F.) for 10 minutes. Tarassuk (1939) found it to be completely inactivated at 54.4°C. (130°F.) for 30 minutes. Krukovsky and Herrington (1942) heated fresh cream to 170°F. and cooled it



immediately. No appreciable lipolysis occurred in butter samples churned from this cream.

(b) Cheesiness

Hammer concluded that the action of bacteria in the butter is responsible for cheesiness although natural protease is present in milk and likely to be present in the cream.

P. putrefaciens appears to be a common, causitive species. Spitzer et al. (1927) added various proteolytic bacteria to pasteurized cream which was churned into butter. Cheesiness was one of the off flavors that appeared in the butters. Hietaranta (1949) noted that many of the bacteria present in butter produce in milk and cream cultures a cheesy flavor. In butter this flavor defect is less frequent but it can be considered to be caused by the decomposition of proteins. Hammer is in agreement with this conclusion.

Rogers (1912) reported that with flash pasteurization protease presumably from milk was much weakened by temperatures between 71°C. (159.8°F.) and 77°C. (170.6°F.) but was not destroyed at 93°C. (199.4°F.), the highest temperature employed. As judged by the production of soluble nitrogen compounds, the data of Spitzer et al. appeared to indicate that pasteurization of cream (148° to 155°F. for 20 minutes), containing proteolytic enzymes elaborated by bacteria, did not necessarily destroy the enzymes. These results would tend

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to indicate that the natural protease of milk and the bacterially induced protease of cream are heat resistant.

(c) Oxidation

White (1944) considered that oxidation is one of the most important causes of butter deterioration during storage and he regarded it as the direct cause of off flavors such as stale, storage, oily, fishy and oxidized.

Bailey (1951) in his review of the mechanism of oxidation reported the initial step to be the addition of oxygen at or near the double bond to form an unstable compound, generally designated as a peroxide. During the early stages of oxidation (induction period) the peroxides appear to be relatively stable so that the quantity present is more or less parallel with oxygen absorption. In the later stages they begin to decompose or react with one another or with other products of oxidation to produce the compounds actually responsible for the oxidized flavor.

Although oleic acid oxidizes much less readily than the more unsaturated acids, Bailey, nevertheless, thought it probable that the flavor resulting from true oxidation is contributed largely or entirely by oleic acid. Holm and Greenbank (1924) observed that the methyl and ethyl esters of oleic acid readily acquired an oxidized odor with limited oxidation, whereas pure esters of linoleic and linolenic



acids developed relatively little odor after absorption of large amounts of oxygen and never developed an odor bearing much resemblance to that of typically oxidized fats.

Bailey suspected that the readily formed peroxides of linoleic and other polyethenoid acids catalyze the oxidation of oleic acid and thus contribute to poor flavor stability.

While Deatherage and Mattill (1939) found that oleic acid oxidized to form peroxides, peracids, aldehydes, substituted ethylene oxides, acids, alcohols, combinations of these and water, Powick (1923) concluded that the flavor is chiefly due to the presence of aldehydes of medium molecular weight, particularly heptylic and nonoic.

Lea reported that freshly made butter contains an average of 5% by volume of entrapped air and the oxygen of this air slowly disappeared during storage. Barnicoat (1948, b) concluded that in addition to the oxygen present in free air, there presumably is sufficient loosely bound oxygen present in butter, associated with olfinic linkages of the fat, phospholipids and other lipids as well as with the protein, to allow incipient oxidation changes to proceed at an unimpeded rate during storage.

If oxidation flavors do not show up on stored butter and particularly if other flavors do appear, there is some reason to suspect that the growth of microorganisms has



impeded oxidation. Hunziker (1940) reported that workers found the growth of bacteria in cream contaminated with copper salts retarded the development of tallowy flavors. The conclusion was that the oxygen was utilized by the bacteria.

In the oxidation of fats, the conclusion of many workers is that certain of the heavy metals are very efficient catalysts. Lea observed that the effectiveness of a particular metal or alloy in accelerating oxidation in the fat of foods brought into contact with it will depend upon the following factors: (a) the possibility of a catalyzed oxidation of the fat at the surface of the metal without appreciable solution of the latter, (b) the ease with which the metal is attacked and dissolved under the conditions of use, and (c) the magnitude of the effect of the metal once it has been brought by solution into contact with the fat. Bailey came to the conclusion that the catalysts most likely to be encountered in commercial fat are metallic soaps formed through the action of free fatty acids on metallic equipment.

Lea reported that salts of copper, iron, nickel, cobalt, chromium and manganese have been found to produce tallowy flavors when incorporated into butters at concentrations of from 2 to 50 p.p.m. Copper was much more effective than any of the other metals, 2.0 p.p.m. being sufficient to produce an incipient tallowy flavor after storage for six days at room temperature. Tin and aluminum were inactive at



a concentration of 100 p.p.m. Pont found that copper contamination even in the low range of 0.06 to 0.17 p.p.m. was important, being correlated with peroxide and aldehyde values and final grades after the butter had been held three months at room temperature. Perrin et al. (1951) concluded that a copper content of over 0.15 p.p.m. and an iron content of over 1.50 p.p.m. in butter was not satisfactory.

In the absence of heavy metals, the oxidation of butterfat will be accelerated if the fat is subjected to strong light. Stebnitz and Sommer (1937) concluded from their data that all sources of visible and ultraviolet light are effective in catalyzing the reaction. Ultraviolet radiations were more effective than radiations of greater wave length. The intensity of the light also influenced the speed of oxidation. They found that ultraviolet rays and strong sunlight practically eliminated the induction period while diffused daylight acted much less efficiently. Infrared radiation did not act as a catalyst. These results are in agreement with those of Mukherjee (1950, a).

Barnicoat (1948, c) observed that with butter of low copper and iron content, exposure to weak, interior, diffuse daylight (not exceeding two hours) had no deleterious effects on the product. However, prolonged exposure (4-24 hours) at low light intensity or short exposure to the sun's rays through glass tended to lower the quality of the butter.



The effect of temperature according to Brown and Thurston (1940) is important only as a regulator of the rate of oxidative change, the rate being approximately doubled per  $10^{\circ}\text{C}$ . ( $18^{\circ}\text{F}$ .) increase. Lea noted that oxidation in the absence of light and positive catalysts has a normal temperature coefficient, but that temperature becomes progressively less important as the intensity of illumination or the content of active metal increases.

Other substances have also been classed as pro-oxidants among which is sodium chloride or common salt. Hills (1949) reported that traces of magnesium chloride were responsible for accelerated oxidation when finely ground commercial salt was dispersed in butterfat. He found that a solution of salt dispersed in butterfat also accelerated oxidation, particularly in the presence of acid. This phenomenon was caused by the formation of free chlorine which accelerated the oxidation of the fat.

Some evidence has been obtained to show that diacetyl also has pro-oxidant properties. Ritter and Nussbaumer (1939) observed that peroxide number determinations made on samples of butterfat which were heated to  $104^{\circ}\text{C}$ . ( $219.2^{\circ}\text{F}$ .) for eight hours indicated that the addition of 0.1 to 1% diacetyl resulted in a significant increase in peroxide number. However, Barnicoat (1937) found that the addition of 4 to 6 p.p.m. of diacetyl to butter stored for three and a



half months at 14°F. was not important in promoting oxidative deterioration of the butter.

Mukherjee (1950, b) observed that the development of free fat acidity in butterfat proceeded only in the presence of moisture. Peroxide formation increased concurrently. He suggested that moisture in bringing about the accelerated fat hydrolysis provides free fatty acids which are more easily oxidizable than the glycerides. According to Greenbank (1949) low moisture retards oxidation.

The induction period of fat preceding the onset of autoxidation has been related to substances called anti-oxidants which are naturally associated with fats. The presence of antioxidants provides a protection of the ethenoid structure against oxygen and peroxides. Antioxidants added to fat or fat containing food must be nontoxic, palatable, dispersible and relatively cheap in manufacture.

Mattill (1945) classified the compounds exerting an antioxygenic influence as: (1) with few exceptions, the only substances with primary action are ortho and para, di and polyphenolic compounds or substances having a similar electronic configuration; (2) all other substances which delay or inhibit the autoxidation should be called synergists because they merely reinforce the effect of phenolic compounds and have little activity apart from them. Lea (1952) pointed out that others function by forming inactive complexes with traces of pro-oxidant metals.



Mattill (1931) observed that the antioxygenic capacity of phenols resides in two hydroxyl groups in the ortho or para configuration; in the meta position the compound is inactive. The hydroxyls are ineffective unless they are attached directly to the ring.

Golumbic (1943) noted that when tocopherol acts as an antioxidant it is not merely being oxidized to tocoquinone but at the same time it reduces the active peroxides and, therefore, breaks the reaction chain that would otherwise be started. As long as tocopherols are present, peroxides do not accumulate in amounts sufficient to cause oxidative formation of the secondary products that characterize oxidation.

However, the action of antioxidants is not clear cut because their action varies in different fat systems. Hilditch (1944) noted that work done has demonstrated that the order of efficiencies of a selected group of antioxidants may differ widely according to whether they are examined in relation to anhydrous fats or to fat emulsions of varying concentration.



### Modification of Butter

Pont (1945) reported that in experiments dealing with the keeping quality of butter at summer room temperatures bacterial defects were controlled by the use of boric acid, a preservative, or by a process of vacuum working combined in each case with salt concentrations ranging from 2.5 to 3.0% or more. Barnicoat (1948, a), however, found the addition of 0.125 to 0.5% of borates, calculated as boric acid, to unsalted sweet cream butter gave no consistent improvement to its keeping properties at various temperature levels of storage (14° to 65°F.). In comparison with the control butters, salt and the borates did not retard deterioration. At higher temperatures borates appeared to promote fishy flavors and oxidation of the fat. Reinart and Brown (1953) observed that maintaining 15% salt (in the serum) and heat treating the product at 180°F. for 20 minutes caused the bacterial activity to be small.

Whereas Pont noted that vacuum working the butter appeared to control bacterial defects, he observed that the rate and extent of fat oxidation in tinned butter was not affected by a reduction of the air content in vacuum working. In normal butter the air content by volume was found to range from 3.72 to 6.52% while that in vacuum



worked butter ranged from 0.35 to 1.38%. Barnicoat (1948, b) found that the keeping properties of butter of sweet cream or of the milk starter type were not dependent on the free air content which he found to vary from 4.9 to 6.7%. Barnicoat noted that these values were slightly higher than those of other workers, the averages of which ranged from 3.95 to 5.37%.

Among the substances which have been found to inhibit oxidation of dry butterfat, phenolic and polyphenolic compounds are of considerable importance, but as Swartling (1949) noted, very few of them have been tested in butter. He surmised that the special structure of butter makes it impossible to draw any conclusions with regard to butter from work on pure butterfat. While Swartling found hydrocaffiate, ethyl gallate and nordihydro guaiaretic acid (N.D.G.A.) to be the most effective of the substances tested, the tests covered only a short period of time. Pont noted that ethyl gallate at the concentration of 0.02% in tinned butter showed an antioxidant effect judged by low peroxide values and the absence of pronounced tallowy flavors, but deleterious flavor changes still occurred.

Dahle and Josephson (1937) observed that the addition of the aqueous extract of oat flour to sweet or sour cream



prior to pasteurization and churning materially improved the keeping quality of the resulting butter over a two month storage period at 40° to 45°F.

Barnicoat and Palmer mentioned that soluble phosphates and citrates have been proved to be effective antioxidants in cream used for buttermaking. Later, Barnicoat (1948, a) found that the use of citrates and phosphates in butter was not very effective.

Reinart and Brown observed that skim milk powder added to a butter spread at the rate of 2.5 to 3% increased the stability of the butterfat against oxidation. Vitamin C at the rate of 0.015 to 0.02% was also found to be effective.

During World War II the American armed forces were in need of a spread for bread to be used when refrigeration facilities were unavailable. Remaley et al. (1949) reported that a modified butter spread, Carter's spread, was developed. The modification included: (1) the addition to freshly churned creamery butter of enough cottonseed oil flakes to raise the melting point of the finished product to 110°F. and (2) heat treatment of the mixed ingredients for at least 10 minutes at temperatures between 150° and 180°F. While the final product remained emulsified at tropical temperatures, accelerated tests showed that oxidized and eventually a rancid flavor developed after 60 days at 110°F. Off flavors



appeared between six and nine months at room temperature or between four and six months at 100°F. After five months' storage at 90° to 100°F., some samples were found to have a butyric acid flavor characteristic of aged butter. No improvements were noted by processing the product at higher temperatures.

The rate and degree of flavor deterioration in butter is advanced by increasing the temperature of storage. During World War II in the Pacific theatre, normal butter deteriorated rapidly, especially at temperatures above its melting point. As Pont pointed out under this condition the butterfat and serum separate into layers and the normal physical restrictions on bacterial growth are no longer operative.

#### The Development of a Butter Concentrate

Wiley and Coombs reported that research carried out by the Australian Council for Scientific and Industrial Research revealed that butterfat could be prepared which had good keeping qualities when stored at atmospheric temperatures. Coombs (1949) noted that a moisture level of 0.2 to 0.3% was not sufficient to support bacterial activity. Wiley and Coombs reported that while dry butterfat with added salt, stored in one pound cans, was not subject to bacterial



deterioration, it did show signs of tallowiness after two weeks. Samples without salt were not oxidized. However, the butterfat was too soft for hot climates.

Attempts were then made to raise the melting point of the fat. Although it was found that 105°F. was the highest melting point consistent with palatability it was noted later that at a melting point of 102°F. the flavor was improved because of the absence of a slight greasiness on the palate, which registered at 105°F.

The effect of oxygen in the oxidation of fat has long been recognized. Lea et al. (1943) incubated samples of butterfat in the presence of varying amounts of oxygen at 100°C. (212°F.). Their experiments indicated that at or above an oxygen concentration of 0.05 ml. per g. of fat, the fat showed definite tallowiness after 50 hours.

Schaffer et al. (1946) found that with milk fat samples stored at 30°, 50° and 70°C. (86°, 122° and 158°F.), after prolonged storage, only those samples in the presence of less than 0.009 ml. of oxygen per g. of fat (0.80% by volume) did not develop a tallowy flavor. A concentration of 0.013 ml. per g. of fat (1.19% by volume) produced a definitely oily and tallowy flavor after 16 weeks at 50°C. (122°F.). Shipstead and Tarassuk noted that to prevent oxidative deterioration in dehydrated whole milk the free oxygen level in the container has to be lowered to or below 0.01 ml.



per g. of powder. Mukherjee (1950, c) observed that oxidation slowly proceeds in butterfat stored in an inert atmosphere, although the induction period is nearly doubled. This information points to the fact that oxidation can be brought about by small amounts of oxygen dissolved in the fat.

Coombs reported that in producing butter concentrate most of the process is under vacuum. Close attention was given to the cans and to the seaming machine. Unless the seam was perfect the oxygen content of the product rose to 2% by volume in seven days. Wiley and Coombs reported that the air content of butter concentrate was reduced to the order of 0.25% by volume.

Hills concluded that the pro-oxidant effect of the common salt came from traces of magnesium chloride which it contained. The problem was overcome by finely grinding the salt in contact with a small amount of anhydrous sodium carbonate.

In developing the butter concentrate the solids—not fat were replaced by skim milk powder which was found to possess antioxygenic properties. The optimum amount of powder reported by Coombs appeared to be two parts per one part of salt.

In the problem of producing the butter concentrate, attention was given to heavy metal contamination. The first step in the production was to free the butteroil from the



serum which holds the metals. McDowall et al. (1943) reported that a dry butterfat could be produced which contained 0.01 to 0.03 p.p.m. copper and 0.02 to 0.15 p.p.m. iron. Coombs noted that the copper contamination in skim milk powder used in the concentrate could be almost eliminated by manufacturing the powder in all stainless steel spraying plant. Hence, by starting with suitable materials and by using stainless steel equipment a butter concentrate could be produced which was very low in metals.

### EXPERIMENTAL

#### I. METHODS

##### (a) Source and Quality of Butter

First grade creamery butters with flavor scores of 39 or 40 and laboratory churned butters were used in this investigation, with the exception of butter concentrate 4 in which butteroil produced by the Cherry-Burrell continuous buttermaking equipment was used. The laboratory churned butter was made from cream produced to minimize its content of microorganisms and also its contamination by copper and iron. All the metal equipment used in processing the butter in this research was constructed of stainless steel, the only exception being the enameled tinned cans in which the butter was packed.



(b) Modification of Processing and Composition

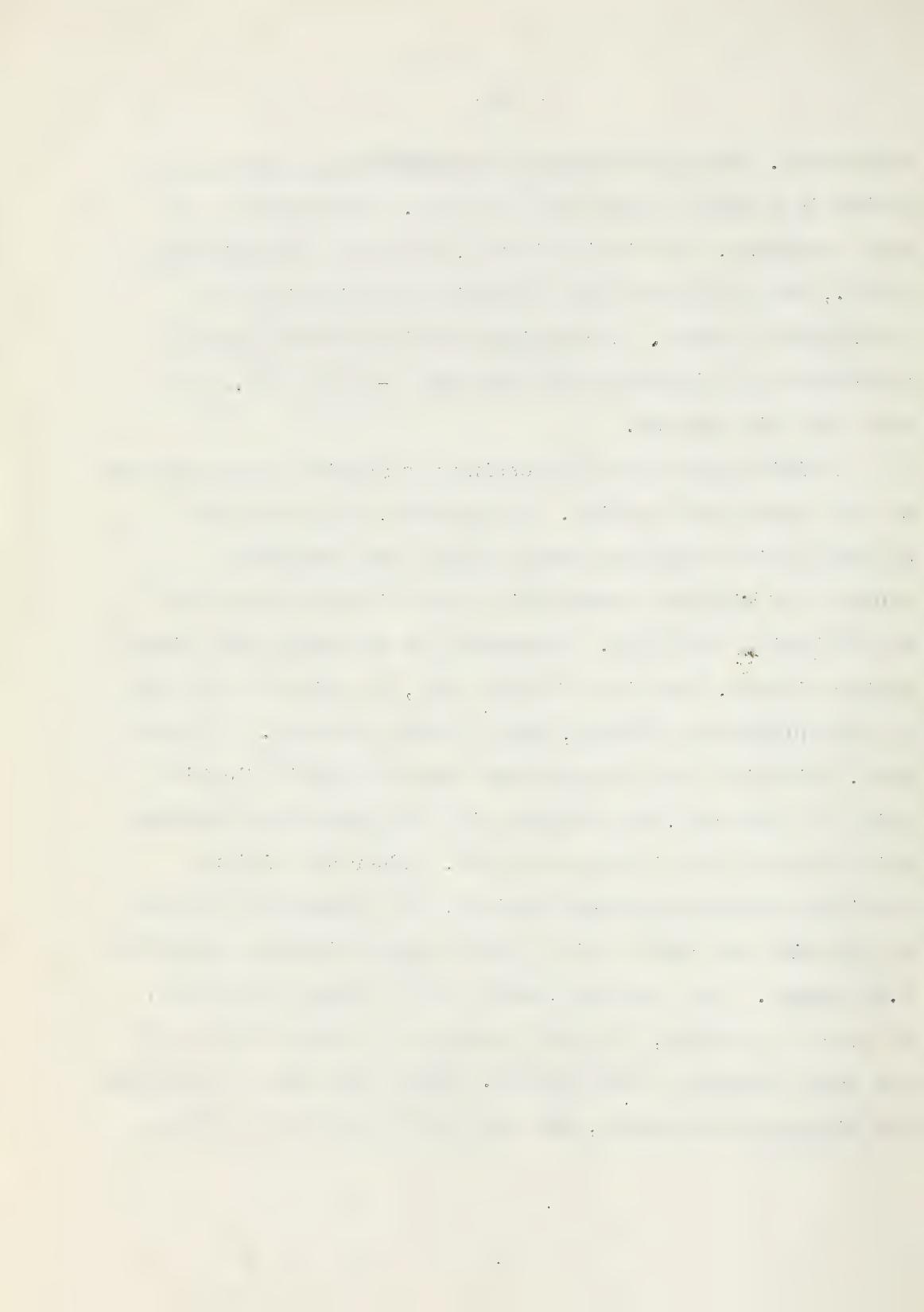
Only in the preliminary investigation were the butters put directly into cans without further processing. Because smaller cans were not available at the initiation of the investigation, previously sterilized, enameled 401 x 212 cans were used for butters 1 to 5. One-quarter pound pats of these butters were wrapped in parchment prior to the canning. Butters 18, 19, 31 and 51 were packed, without headspace, into size 304 x 211 enameled cans which were used in all of the experiments except as noted above. The cans were sterilized before use.

The results of the preliminary investigation indicated that changes in the processing and composition of the butter were necessary to increase its flavor stability. Consequently, the following changes were made in the first modification. The butters were melted and heated to a temperature of 140°F. After adjusting the composition (16% water and 2% salt, except when unsalted butter was used) and incorporating any additives, approximately 5 oz. of the liquid mixture were dispensed into sterilized enameled 304 x 211 cans, homogeneity being maintained by thorough mechanical stirring. After sealing, the cans were heat treated at a temperature ranging from 170° to 180°F. for 10 minutes in an agitated



water bath. The temperature was measured by a thermometer sealed in a water filled 304 x 211 can. Subsequent to the heat treatment, the liquid mixture, cooled to approximately 100°F., was emulsified and solidified by agitation in a refrigerated shaker. A centrifuge equipped with a shaker attachment and placed in the cold room (-5° to -8°F.) was used for this purpose.

A similar method of processing was applied to the butters of the second modification. In addition, duplicate cans of each modified butter (both creamery and laboratory churned) of different composition were nitrogen packed by the following technique. Subsequent to dispensing the liquid butter mixture, the lids of those cans, in which the air was to be replaced by nitrogen, were loosely attached. A vacuum oven, maintained at a temperature above the solidification point of butterfat, was employed in the evacuation procedure which removed the air from the cans. After the minimum possible pressure had been attained, the vacuum was released by flooding the chamber with premium grade nitrogen containing 0.2% oxygen. This pressure, which varied from 16 to 30 mm. of mercury absolute, could be lowered no further because of the water content of the butters. After the second evacuation and nitrogen replacement, the cans were immediately sealed.



The temperature used in the heat treatment of the butters of the second modification was 180°F.

The butter concentrate was processed similarly to the nitrogen packed modified butters except for the following differences. In the first stage of processing, after the butter had been melted, the butteroil was decanted from the serum and run through a cream separator twice to reduce the residual serum to a minimum and thus minimize the water content. Another difference in the method of processing was that some of the butter concentrates were packed in an atmosphere of nitrogen which had been passed through a chromous tower to remove the residual oxygen. The pressure was lowered to approximately 6 mm. of mercury absolute during the evacuation procedure.

Table 2 shows which of the butters of the second modification were nitrogen packed and Table 3 shows which of the butter concentrates were packed with washed nitrogen.

#### (c) Additives

The composition of the modified butters and butter concentrates is shown in Tables 1 to 3.

The Tenox II, incorporated in the various modified butters, was composed of 20% butylated hydroxyanisole,



4% citric acid and 6% propyl gallate dissolved in propylene glycol. The N.D.G.A., with one exception, was added as a mixture which contained 10% N.D.G.A., 5% citric acid and 85% propylene glycol. To butter 16, N.D.G.A. was added in powder form and at a rate of 0.1%.

Because a bland, hardened oil was not available when the second modification was initiated, it was necessary to refine and deodorize hardened cottonseed oil in the laboratory. This oil was not completely bland but the remaining flavor was considered to be insignificant. A bland, fully hydrogenated cocoanut oil became available in time to be incorporated in the first butter concentrate. Unfortunately, the melting point of this product was too low to be effective. Later, a fully hydrogenated peanut oil was obtained and used in the remaining butter concentrates.

Salt (sodium chloride) added to the butter concentrate was ground in a ball mill in the presence of  $1\frac{1}{2}\%$  anhydrous sodium carbonate and subsequently sifted through a silk screen of approximately 140 mesh per inch.

The spray processed skim milk powder incorporated in the butter concentrate was manufactured in stainless steel equipment to keep heavy metal contamination at a minimum.

Diacetyl (C.P.) added at an approximate concentration of 4 p.p.m. was incorporated in two butter concentrates.



(d) Storage Temperatures

Room temperature and 95°F. as well as 0° and 45°F. were the levels of temperature utilized in the storage of the canned butters of the preliminary investigation and first modification. The room temperature storage (60° to 80°F.) was used to simulate actual storage temperatures to be encountered in the use of the product. The two temperatures used in the storage of the butters of the second modification and of the butter concentrate were 70° and 90°F.

(e) Testing Procedures

When samples for the plate count were taken, care was exercised to avoid contamination upon opening the cans. Immediately after this step, the product was tested organoleptically. Following flavor scoring the butter or butter concentrate was melted and the butteroil decanted

and centrifuged at 800 r.p.m. for 10 minutes to separate any serum remaining in the oil. To remove the particles of skim milk powder and/or salt which were still suspended in the oil from the butter concentrate after centrifuging, it was necessary to pass the butteroil through glass wool. The butteroils were used for the chemical tests. When the



tests measuring fat oxidation could not be applied immediately after the preparation of the butteroil, samples of the oil were weighed into flasks which were then covered and placed in the cold room until the determinations could be made.

The plate count, used to estimate the bacterial population in the butters of the preliminary investigation and first and second modification, was similar to the standard plate count for bacteria outlined by the American Public Health Association (1948) except for an incubation temperature and time of 25°C. and five days. Difco malt agar acidified to pH 3.5 was used as the medium for the yeast and mold count. These plates were also incubated at 25°C. for five days.

Hydrolysis of the butterfat was followed in all of the butters and butter concentrates by employing the free fat acidity test, using the method reported by Dunkley and Wood (1943). The results are expressed as ml. of 0.05 N NaOH required to neutralize the free fatty acids in a 5.0 g. sample of the butterfat.

The iodometric method of Wheeler (1932) was used to determine the peroxide content in all of the samples. A ferric thiocyanate procedure for determining peroxides, as outlined by Chapman and Mackay (1949) was also employed in analyzing the butter concentrate samples. The results of both methods are expressed as milliequivalents per kg. of fat.



An acidified solution of thiobarbituric acid when heated with oxidized milk fat has been found to form a red color. The thiobarbituric acid test, as developed by Patton and Kurtz (1951) to measure fat oxidation, was applied to the samples of the second modification. The intensity of the developed color was determined on 10 ml. aliquots of the reaction mixture by an Evelyn colorimeter, using a 540 millimicron filter. The results are expressed in terms of absorbance in which absorbance is equal to the log base 10 of the reciprocal of the transmittance of light. Biggs and Bryant (1953) found that oxygen in the reaction flask influenced color development in the T.B.A. test. In the absence of oxygen the color development was more closely related to the development of an oxidized flavor. Their method was applied only to the butter concentrate and it was modified by refluxing a 4 g. sample of butterfat with the reagent mixture for 45 minutes. The intensity of the color formation was determined on 6 ml. aliquots of the solvent layer, using the Evelyn colorimeter with a 540  $\mu$  filter.

Copper and iron concentrations in the butters of the second modification were estimated by a filtration method developed by Perrin et al. (1951). The amounts of solvents suggested were doubled and the intensity of color in 6 ml.



aliquots of these solvent layers was determined by the Evelyn colorimeter, using a 440  $\mu$  filter for copper and a 490  $\mu$  filter for iron.

The protein content of the butter concentrate was approximately four times as great as the amount present in the butters and apparently interfered with the extraction of the metals. Accordingly, a wet ashing technique similar to that outlined by De Ath et al. (1946) was used for the estimation of copper and iron in the concentrates. The methods for development of color and measurement of the intensity of the developed color were similar to those used in the filtration technique.

The method used for determining the water content of butter concentrate 1 was that recommended by the American Oil Chemists' Society (1946) for the determination of moisture and volatile matter. A Karl Fischer titrimetric procedure taken from Mitchell and Smith (1948) was used to determine the water content in the other butter concentrates.



## II. RESULTS

### A. Preliminary Investigation

#### (a) Growth of Microorganisms

While the results of the bacterial plate count indicated that at a storage temperature of 0°F. no growth of bacteria occurred in the canned butters, Table 4 shows that there was a definite increase in the bacterial number at the higher storage temperatures. In each of the five butters stored at 45°F. the population did increase during the course of storage, particularly in butter 4 which contained no salt. It can be seen in Table 4 that the growth of bacteria in the butter appeared to be greater at room temperature storage than at either 45° or 95°F. storage. Fig. 1 which represents the average bacterial numbers of the nine butters indicates that at room temperature the bacteria grew rapidly and reached a maximum population, as shown by the plate count, after approximately one month's storage. Thereafter, the bacterial number declined quickly. The results of the bacterial counts in the butters stored at 95°F., as well as in butter 20 stored at room temperature, indicate that a maximum population may have been attained before the end of one month's storage.



The results of the yeast and mold counts, not presented here, show that at 45°F. storage yeast growth was quite abundant while mold growth occurred in only two butters. In general, at room temperature yeast growth appeared to be slight while mold growth was extensive. There was little evidence of either yeast or mold growth at the 95°F. storage.

Although the canned butters stored at 0°F. showed no evidence of an increase in free fat acidity, Table 5 indicates that similar butters stored at the higher temperatures did increase in value, indicating that hydrolysis of the fat was occurring. Table 5 reveals that the increases in free fat acidity value in those butters stored at 45°F. were less than the increases in duplicate butters held at room temperature. Fig. 2 based on the average free fat acidity values of nine butters stored at room temperature shows the rapid increase that occurred at that level of storage. Four of the nine butters displayed a relatively slow increase in the acid value and the bacterial growth in these butters was relatively small. Exceptionally high values were obtained in butter 5, considering the rather low plate count. However, it is probable that a bacterial population may vary in its percentage of lipolytic organisms. Table 5 indicates that in general, the increases in free fat acidity in the butters



stored at 95°F. were not as large as in the corresponding butters held at room temperature. Possibly, the lipolytic organisms present were not as active at 95°F. as at room temperature.

(b) Development of Off Flavors

At a storage temperature of 0°F. the canned butters were not subject to flavor deterioration during the four months they were under observation. However, flavor scoring revealed that off flavors developed on the butters stored at 45°F. as early as the end of the second month. By the end of the fourth month the off flavors, which included rancidity, were pronounced on all five butters. In general, off flavors were definite after one month in those butters stored at room temperature. By the end of the third month, seven of the nine butters contained a pronounced rancid flavor. Butter 21 showed an exceptional flavor stability. While a variety of off flavors appeared on the butters stored at 95°F. within one month of storage, the predominant off flavors were cheesy and rancid. A comparison of the organoleptic evaluation and the free fat acidity values revealed that a rancid flavor appeared at no constant fat acidity value. However, in general it can be said that when a butter yielded a value of 1.50, it was rancid organoleptically.



Table 6 indicates that peroxide values appeared only a few times on the canned butters stored at 45°F., room temperature and 95°F. Although it is apparent that butter 3 was susceptible to oxidation at the three temperature levels, an oxidized flavor was distinguishable in only a few instances in that butter. Butter 20 stored at room temperature was the only one to yield consecutive peroxide values and to be judged tallowy over the four month period. The bacterial counts and free fat acidity values were comparatively low in butter 20. In general the flavor scoring results indicated that fat oxidation was of less importance than fat hydrolysis in contributing to the low flavor stability of the canned butters.

#### (c) Physical Stability of the Butter

Although results indicated that the canned butters retained their normal physical stability at 45°F., the majority of the butters at room temperature storage were oiled off by the end of the fourth month. At 95°F. oiling off had occurred in all of the butters under observation by the end of one month. On advanced storage at 95°F., the oiling off became more severe and resulted in a separation of the aqueous and fat phases in some of the butters.



B. First Modification

(a) Growth of Microorganisms

The initial plate counts shown in Table 7 indicate that the heat treatment was quite effective in reducing the bacterial population in these butters. This table also shows that, with the possible exception of two unsalted butters (8b and 9b) which exhibited a few isolated high counts, little or no bacterial growth occurred in the butters of laboratory source (8a to 16b) stored at 45°F. With the exception of 15b, the bacterial content of butters 12a to 15b (creamery source) appeared to decrease in number over the storage period at 45°F. It can be seen that at room temperature four butters (8a, 8b, 9a and 9b) which did not contain the preservative, sodium benzoate, displayed a large increase in bacterial number within the first month. The other butters of laboratory origin which contained sodium benzoate showed little or no growth. In some of the butters of creamery origin the number of bacteria appeared to increase, regardless of the presence of the preservative. However, this increase was not as great when salt was present. Results of the bacterial plate count in the two butters stored at 95°F. are not presented here. The results obtained indicated, though, that the bacterial population increased rapidly at that temperature in spite of the presence of sodium benzoate and salt.



The results of the yeast and mold count showed that the heat treatment given the modified butters was effective in preventing the growth of any of these organisms which were present originally.

An experiment designed to find the effect of separation of the fat and serum phases of a modified butter upon bacterial growth, both in the presence and absence of salt, was included in this section. Half of the butters were left unchurned subsequent to the heat treatment. Storage of these butters was at room temperature. Figure 3 indicates that a separation of the fat and serum phases resulted in a rapid increase of the bacterial population, regardless of the presence of salt. However, the increase was considerably greater in its absence. Growth of the organisms was restricted by emulsification of the two phases and apparently even inhibited in the presence of the 2% salt.

Values of the free fat acidity test recorded in Table 8 show that when stored at 45°F. only one butter, 8b, increased definitely in value and concurrently bacterial growth occurred in this butter. At room temperature only one butter, 8b, showed a large increase in value and this was accompanied by a considerable increase in the bacterial number. Small increases noted in the free fat acidity values of other butters stored at room temperature also were accompanied by increases in bacterial content. Free fat acidity values steadily increased on the two butters stored at 95°F.



At both 45°F. and room temperature slight fluctuations occurred from month to month on those butters which were not increasing in free fat acidity value. These fluctuations may have been caused by variations in the individual cans.

No definite phenolphthalein end point could be obtained in the free fat acidity determination on butters 16a and 16b. The presence of 0.1% N.D.G.A. is believed to have been responsible for the interference.

(b) Effect of the Emulsifier

While there was no breakdown of the butter emulsion at 45°F., eight of the 16 butters which contained an emulsifying agent showed evidence of oiling off, when stored at room temperature. Oiling off was extreme by the end of the first month in the two of these butters incubated at 95°F. The results would indicate that at a concentration of 0.2% the mixture of mono- and diglycerides are not effective in preventing oiling off at room temperature or above.

(c) Results of the Peroxide Test

Table 9 shows that a peroxide value was obtained on only one butter (8a) at 45°F., although the peroxide test was not applied to the majority of butters after the third month



of storage. At room temperature no value had been recorded on any butter by the end of the first month, but by the end of the second, third and fourth months, peroxide values had appeared on nine of the 16 butters. Concentrations of peroxides sufficiently high to be registered by the determination, may have been present in those butters not tested at the end of the fourth month. No peroxide values were apparent on the two butters stored at 95°F.

(d) Flavor Stability of the Butter

Flavor scoring which can be observed in Table 10 indicated that off flavors developed in only four of the butters stored at 45°F. The one butter (8b) in which a rancid and bitter flavor became noticeable also increased in its free fat acidity level. Tallowy flavors were evident on butters 8a, 9a and 15a by the end of the fourth month. At room temperature storage the organoleptic evaluations (shown in Table 10) indicate that in the majority of the butters slight off flavors had been detected by the end of the first month. After three months of storage the off flavors had become pronounced. Although tallowy flavors did become evident after one month of storage in the two butters at 95°F., a cheesy flavor developed on advanced storage.



Excluding flavor deterioration in butters 8a and 8b, which was no doubt bacterially induced, fat oxidation was apparently responsible for the predominant off flavor at room temperature. A slight tallowy flavor became noticeable as early as the first month on eight butters and by the end of three months this flavor was pronounced in 13 of the 16 butters. Neither the presence or absence of salt nor the presence of antioxidants seemed to influence greatly the fat oxidation.

(e) Texture of the Modified Butter

The texture of the modified butters, when examined at the lower storage temperature (45°F.), was coarse and somewhat brittle. A slow rate of cooling during the emulsification stage of processing of the modified butters was believed to account for the formation of large fat crystals which would cause the coarse, brittle texture.

C. Second Modification

When bacterial growth was slowed down or inhibited in the butter, as indicated in the foregoing experiments, a tallowy flavor became detectable and increased in intensity with time. In order to delay or eliminate the development of this flavor defect a second modification was initiated in which butters were nitrogen packed.



As an introductory investigation two modified butters from the same lot of creamery butter were nitrogen packed and stored at both 70° and 90°F. The composition of butter 18 was not altered but both N.D.G.A. and sodium benzoate were incorporated in butter 19. Results of the bacterial plate count, not presented here, indicated that while there was no increase in the number of bacteria at 70°F. storage, growth did occur during the first and second months in both butters at 90°F. Figure 4 shows that both butters at 70°F. displayed a slow but fairly steady increase in free fat acidity. The increase, however, was considerably greater at the higher storage temperature.

According to Figure 4, detectable peroxide formation occurred after one month of storage in butter 18 which contained no additives. An increase in the T.B.A. absorbance value corresponded with this peroxide formation, but, decreased in magnitude after the fourth month. At the end of the fourth month the flavor was definitely tallowy. Although butter 19 yielded a small peroxide value at the end of the fifth month, definite peroxide formation was not detected until the tenth month. At this time the T.B.A. absorbance value increased considerably. A distinct tallowy flavor, as well as a black discoloration were characteristic of butter 19 after 10 months



of storage. Neither of these butters showed evidence of peroxide formation or change in the T.B.A. absorbance value during their storage at 90°F. However, a cheesy flavor was noticeable on both butters after the second month.

In view of the generally favorable results from butter 19 stored at 70°F. the investigation was expanded with further slight modifications of composition being included. The butters used in this experiment came from two sources: one lot of creamery butter and one lot of laboratory butter.

#### (a) Growth of Microorganisms

The plate counts shown in Tables 11 and 12 indicate that the heat treatment reduced the bacterial population to the range of 2000 to 4300 in the creamery butters and to 10 or 15 in the laboratory butters. These tables show that little or no growth occurred in the creamery butters stored at the two temperature levels and that the number of bacteria increased only slightly in the majority of the laboratory butters stored at both 70° and 90°F. While in general the bacterial population decreased as the storage period increased, organisms were still present in the butter after the nineteenth month. The determination of bacterial plate counts, as well as the other tests, were discontinued when the butters



had deteriorated sufficiently in flavor to be considered inedible. Although the presence of sodium benzoate appeared to exert little influence on the bacterial numbers in the creamery butters, no growth of bacteria occurred in the laboratory butters containing this preservative.

No evidence of the presence of yeasts or molds was obtained on the butters of the second modification, indicating their complete destruction by the heat treatment.

The free fat acidity values shown in Tables 13 and 14 increased in all of the butters. However, the increase in value was more rapid at the higher storage temperature. By the end of the nineteenth month there were approximately two to three times as much free fat acidity in those butters at 90°F. as in comparable butters held at 70°F. Examples of this trend are shown in Figures 5 to 8. The results of flavor evaluation, which can be seen in Tables 19 and 20, indicate that at 90°F., the laboratory butters were slightly rancid by the end of 11 months while the creamery butters were slightly rancid after 15 months.

The results of flavor evaluation also show that with the exception of 61 which became tallowy and 67, all of the nitrogen packed butters stored at 90°F. developed a cheesy flavor after five to seven months of storage. This cheesy flavor did not become noticeable on butter 67 until the eleventh month.



### (b) Susceptibility Toward Oxidation

The flavor defect results shown in Tables 19 and 20 indicate that with the exception of three butters which became metallic, the non-nitrogen packed butters developed a tallowy flavor after two or three months. Table 15 and Figures 5 to 8 show that peroxides, in sufficient quantities to be detected by the test, had formed in the non-nitrogen packed creamery butters. The nitrogen packed butters of this source were free of peroxide values. A slight tallowy flavor did become noticeable on three of these butters, but, with the exception of butter 33 were not detectable on advanced storage. Table 20 indicates that of the laboratory butters which were nitrogen packed, 53 and 61 became definitely tallowy after three months of storage. Butters 55 and 57 were classed as being slightly tallowy after seven months, but, the oxidized flavor in these butters did not become more pronounced during the interval they were under observation. Table 16 reveals that peroxide values were obtained on all of the laboratory butters which became tallowy with the exception of butter 53. An observation of Tables 15, 16, 19 and 20 indicates that in general the first recorded peroxide value coincided with the appearance of a tallowy flavor. In three instances, however, the oxidized flavor appeared first.



While there was no specific T.B.A. absorbance value which coincided with the appearance of a tallowy flavor, Tables 17 and 18 and Figures 5 to 8 show that generally the value was approximately 0.155 when the oxidized flavor was recognized as being present. The greatest absorbance value did not always occur simultaneously with the first detection of the tallowy flavor. When noticeable fat oxidation did not occur, the absorbance value did increase slightly in magnitude.

While the presence of Tenox II in the non-nitrogen packed butters did not delay the appearance of a tallowy flavor (shown in Figures 6 and 8), the presence of N.D.G.A. did prevent noticeable oxidized flavor development in butters 38 and 58 for about nine months at 70°F. (Tables 19 and 20). However, the presence of N.D.G.A. seemed to be associated with the formation of a metallic flavor and black discoloration of the serum. Incorporation of the antioxidants in the nitrogen packed butters may have been responsible for a somewhat better flavor after 19 months of storage at both 70° and 90°F., although they seemed to give a slight foreign flavor to some of the butters.

Corrosion of the exposed metal sections of the enamel-lined cans of nine different non-nitrogen packed butters occurred during the time they were in storage. It is believed



that if the non-nitrogen packed butters had been observed over a greater storage interval, corrosion of a larger number of cans would have been evident. Slight corrosion of the cans of only 3 nitrogen packed butters was noticeable over the nineteen month storage period.

The content of oxygen in the cans of non-nitrogen packed butters was calculated to be approximately 0.1 ml. per g. of fat. In the cans of nitrogen packed butters the oxygen concentration was reduced to approximately 0.001 ml. per g. of fat.

Estimations of the iron and copper concentrations of the butters are shown in Table 29. Since the creamery and laboratory butters each came from one lot and since the additives were present in amounts too small to affect the metal concentration, only two nitrogen packed butters of each source were analyzed. The results show that there was little variation in the concentration of each metal in the butters from each source and that no additional metal was added from the cans after storage at 70° or 90°F. The relatively large concentration of iron in the laboratory butters is believed to be the result of rust contamination from one of the stages of processing.



(c) Physical Stability of the Product

With few exceptions those butters stored at 70°F. indicated that the melting point of the fat was sufficiently high to prevent oiling off. The addition of 5% hardened cottonseed oil to those butters stored at 90°F. prevented any breakdown of the emulsion which might result in separation of the serum.

(d) Texture

Because the butters were sufficiently soft at a storage temperature of 70°F., there was no evidence of a coarse texture. However, in the presence of hardened oil the body of the butters was firm enough that a coarse texture was apparent.

D. Butter Concentrate

The data on the second modification indicated that bacteria surviving the heat treatment may have been associated with flavor deterioration in the modified butter. Investigations by Australian workers have shown that bacterial activity can be inhibited by nearly eliminating the moisture content of a butter spread.



In this experiment the study of a product resembling the Australian butterfat concentrate described by Coombs was undertaken. Butter concentrate 1, as a preliminary part of the experiment, was manufactured and placed in storage six months in advance of the other concentrates. Estimation of the bacterial population was not made in any butter concentrate because the composition would have made application of the plating technique difficult without modification.

(a) Moisture Level and Oxygen Removal

Table 3 shows that with the exception of butter concentrates 1b, 1c and 1d the water content was reduced to the range of 0.2 to 0.3% in butter concentrates 1 to 5. A different technique for moisture analysis together with failure to remove all of the curdy material from the butteroil are offered as possible explanations for the higher water content found in the first butter concentrate.

More air was removed from the cans of concentrate than from the cans of modified butter because of the much lower water content. Consequently, the residual oxygen content in the cans of concentrate is lower. By calculation the approximate oxygen concentration is 0.0009 ml. per g. of fat.



(b) Flavor Stability

Exploratory tests indicated that heat treatment of the butter concentrate at 180°F. for 10 minutes did not result in the development of a carmelized flavor. However, a slight carmelized flavor did become evident in the concentrates within the first two months of storage at 90°F. This flavor was not as pronounced in the product stored at 70°F. Only in butter concentrate 1 at 90°F. did the flavor develop in intensity as the storage interval increased. A brown color believed to be associated with carmelization formed in butter concentrates 1b, 1c and 1d between the fourth and sixth month of storage at 90°F. and appeared to become darker as the carmelized flavor became more intense.

The results of the free fat acidity test shown in Tables 21 and 22 indicate that while about half of the concentrates stored at 70°F. for six months increased definitely in value, the majority of comparable concentrates held at 90°F. increased in value. In general, the butter concentrates at 90°F. contained more free fatty acids than replicate butter concentrates stored at 70°F. for six and twelve months. A Comparison of Tables 13 and 14 with Tables 21 and 22 show that the increases in free fat acidity in the butters of the second modification were much greater than in the butter



concentrates stored at the same temperature for a similar length of time. No rancid flavor became noticeable in the concentrates.

According to the results of the iodometric peroxide test shown in Tables 23 and 24, detectable peroxides developed in three butter concentrates: 1a stored at 90°F., 7 and 8 stored at both 70° and 90°F. The results of flavor evaluation, not presented here, indicated that a tallowy flavor was evident at the end of the fourth month of storage in concentrates 1a held at 90°F. and in 8 stored at both 70° and 90°F. A very strong diacetyl flavor present in butter concentrates 7 and 8 may have masked a tallowy flavor in butter concentrate 7.

While the ferric thiocyanate peroxide values, shown in Tables 25 and 26, are greater in magnitude than the corresponding iodometric peroxide values, there appears to be some correlation between the determinations since with one exception the ferric thiocyanate peroxide value increased sharply when the first iodometric peroxide value was detected. Although the ferric thiocyanate peroxide value for a given butter concentrate fluctuated from month to month, in general, it remained below 0.05 m.e. if the concentrate did not develop a tallowy flavor. The value for the determination on the butter concentrates which became slightly tallowy was 1.00 m.e. or over before the flavor appeared.



In general, the absorbance value for the T.B.A. determination on the butter concentrates (shown in Tables 27 and 28) remained below 0.080 when no noticeable tallowy flavor occurred. The absorbance value appeared to increase in magnitude in those concentrates which became tallowy in flavor or showed a large increase in peroxide value. In the determinations on butter concentrates 1b, 1c and 1d which had been stored at 90°F. the solvent extracted a yellow color from the reaction mixture. While this color did not appear to interfere with the color intensity determination during the earlier testing periods, it is believed to be associated with the increase in absorbance value at the twelfth month.

According to the results of the iron and copper determinations shown in Table 29, the concentrations of these metals varied only slightly in the different butter concentrates. The average iron content was approximately 0.49 p.p.m. and the average copper content was approximately 0.065 p.p.m.

#### (c) Physical Stability of the Concentrates

No oiling off occurred in any of the samples stored at 70°F. Butter concentrate 1 which contained the hydrogenated cocoanut oil melted during storage at 90°F. and after



approximately six months the emulsion broke. None of the concentrates containing hardened peanut oil showed any sign of oiling off at the higher storage temperature. The palatability of butter concentrates 2 to 8 was not affected by raising the softening point of the incorporated butterfat.

(d) Texture

While the texture of the butter concentrates which had been tempered at 45°F. was rather grainy, the texture of the concentrates held at 70° or 90°F. was quite satisfactory, especially when the presence of suspended solids in the form of salt and milk powder is taken into consideration.

DISCUSSION

The results of the preliminary investigation showed that while the canned butters stored at 0°F. were subject to little change in flavor in the four month period they were under observation, replicate butters held at the higher temperatures of storage deteriorated quickly in flavor. Generally, flavor stability was not maintained beyond one month of storage at room temperature or above. Flavor defect results substantiated by the lack of detectable peroxide values tend to show that fat oxidation was of minor



importance in contributing to the low flavor stability of canned butters. The predominant off flavors to become evident were rancid and cheesy and, no doubt, these flavors were the result of a rapid increase in the number of bacteria in the butter.

An increase in the free fat acidity level in the butters stored at 45°F. or above indicated that hydrolysis of the fat occurred concurrently with bacterial growth and flavor deterioration. A comparison of the results of the free fat acidity test with those of flavor evaluation indicates that a rancid flavor appears at no definite free fat acidity value, but, the true relationship may not be apparent because the determinations were made only at monthly intervals.

The general observation, that the keeping quality of canned butters at storage temperatures as high as 95°F. is inadequate, corroborates the reports of investigators in Canada, Australia and New Zealand. It is essential in many of its uses that a canned butter have a marked stability to flavor deterioration. Consequently, the significance of the data obtained from the first modification, in which changes in composition and processing were employed, are important.



It is evident from the results of the first modification that a heat treatment of 170° to 180°F. for 10 minutes, after sealing the butter in cans, materially reduced the bacterial population and effectively destroyed the yeasts and molds. With few exceptions the remaining number of organisms did not multiply significantly at 45°F. or room temperature and, consequently, there was little definite increase in the free fat acidity in those butters during the period they were under observation. Lack of appreciable bacterial growth also appeared to be associated with the absence, in general, of rancid or cheesy flavors in the butters stored at 45°F. or room temperature.

However, the butters of this experiment, as well as the two initial butters of the second modification, which were stored at 95°F. became severely oiled off, increased in bacterial content and developed a cheesy flavor. These results together with the evidence presented in Figure 3 indicate that when butterfat and serum separate the normal physical restriction on bacterial growth are no longer operative and this may result in serum protein degradation. The data obtained on the use of sodium benzoate as a preservative is so conflicting that it is difficult to arrive at any definite conclusion.



Flavor evaluation revealed that flavor deterioration still occurred in the modified butters stored at room temperature when bacterial growth was insignificant. A tallowy flavor indicative of fat oxidation was definite in all but two butters by the third month of storage at this temperature level. Neither the presence or absence of salt nor the addition of antioxidants, in the concentrations used, appeared to influence the oxidation.

Evidence obtained from the butters of the first modification denoted that in the absence of appreciable bacterial activity, fat oxidation became the limiting factor in the flavor stability of the butter. There was sufficient oxygen available in the cans to permit the formation of a pronounced oxidized flavor. In view of the apparent ineffectiveness of the antioxidants, removal or reduction of the oxygen from the containers was believed to be a possible solution to the problem. Consequently, the second modification involved replacement of the air in the cans with nitrogen. In addition, hardened fat was incorporated in the butter to prevent destruction of the emulsion at a storage temperature of 90°F.

The prevention of serum separation in those butters of the second modification which were stored at 90°F. apparently supplemented the action of the heat treatment and



prevented appreciable bacterial growth. While in general, the bacterial population decreased as the storage period increased, their presence in the butter for at least 19 months may have been associated with fat hydrolysis which continued throughout the period. The increase in free fat acidity which was greater at 90° than at 70°F. was apparently sufficient to cause the rancid flavor. It has been reported by Bailey and Mukherjee (1950, b) that fat in contact with water will hydrolyze in the absence of lipase and that the rate of reaction will become greater as the temperature and acidity increase. In view of the lack of extensive bacterial growth especially at 90°F. storage, hydrolysis of the fat may have been increased by this reaction.

The development of a cheesy flavor in the butters held at 90°F. indicates that some degradation of the serum protein must have occurred. While the bacterial population was at a relatively low level in these butters, sufficient proteolytic organisms may have been present to account for some decomposition of the protein. Reinart and Brown concluded that the presence of a serum salt content of 15% together with a heat treatment for 20 minutes at 180°F. satisfactorily delayed proteolytic bacterial activity in canned butter. However, it should be noted that the serum salt content of the butters of the second modification



was less than 15% and that these butters were heated at a temperature of 180°F. for only 10 minutes. The few published results on the natural protease associated with milk or cream tend to indicate that it is relatively heat resistant. For that reason it is possible that natural protease present in the butter serum was not inactivated by the heat treatment and was responsible for protein degradation.

Rapid development of a tallowy flavor in the non-nitrogen packed control butters shown in Tables 19 and 20 verified the results of the first modification. In general, incorporation of the antioxidants into these control butters, at the concentrations used, provided little apparent protection against flavor deterioration. The absence of oxidative defects in the majority of the butters of the second modification indicates that the oxygen content of the cans can be decreased sufficiently to minimize fat oxidation. The residual oxygen apparently was sufficient, possibly under the influence of an oxidation catalyst, to eventually cause a definite tallowy flavor in 5 of the 16 nitrogen packed butters. The fact that three of these tallowy flavored butters contained no additives, together with evidence that the majority of the butters containing the antioxidants and sodium benzoate did not become tallowy, suggests that these additives inhibited fat oxidation under the conditions of a



reduced oxygen content. However, the antioxidants appeared to give the butters a slight flavor which was foreign to the normal butter. Swartling also observed that N.D.G.A. as well as esters of gallic acid added to butter in effective concentrations gave it a foreign flavor.

Various investigators agree that an iron and copper content in butter below the approximate concentration of 1.50 and 0.15 p.p.m. respectively does not exert an evident catalytic effect in the oxidation of butterfat. With the exception of the iron content in the laboratory butter, concentrations of the two metals in the butters of the second modification are below this level. The presence of a relatively high iron concentration may explain why four of the nitrogen packed laboratory butters became tallowy.

While the results of the oxidation tests were useful in establishing whether or not a particular sample became oxidized their value was confined to supplementing flavor evaluation. The lowest positive value of the iodometric peroxide test was found to be approximately 0.32 m.e., indicating that the determination was rather insensitive to low peroxide concentrations. This factor in consideration with the infrequency of testing may explain why positive values generally did not precede the appearance of a tallowy flavor. Mukherjee (1950, c) and Lea et al. have



found that oxidation of fat in a limited oxygen supply, as in sealed containers, is very much depressed. Their results indicated that a rise to low maximum values in the determinations of various oxidation tests was followed by a rapid decrease in value when the oxygen content of the atmosphere had been depleted. These observations may explain why low maximum peroxide values were obtained in testing the canned butters and also why the values for both peroxide and T.B.A. determinations decreased sharply after the apparent maximum had been attained.

Data on the second modification indicated that fat oxidation could be effectively controlled in the nitrogen packed butters at storage levels of 70° and 90°F. The evidence suggested that these butters could be held for at least 19 months at 70°F. with little flavor deterioration. This is a surprising increase in flavor stability in view of the contention of Pont that canned butter, according to his experiments, could not be modified sufficiently to withstand relatively high storage temperatures. Undoubtedly a butter having flavor stability at a temperature of 70°F. would have many uses and would be much superior to the product in current use. By minimizing butterfat oxidation, the keeping quality of the modified butters stored at 90°F. was also increased. However, at this temperature level, it is



apparent that fat hydrolysis and possibly serum protein degradation becomes the limiting factor in flavor stability. The development of rancid and cheesy flavors in the nitrogen packed butters of the second modification prompted the investigation of the keeping quality of a butter concentrate. It was believed that by reducing the serum content to as low a level as possible flavor deterioration caused by these two off flavors possibly could be eliminated.

No rancid flavor became apparent in any butter concentrate during the time it was under observation. However, since the free fat acidity level did increase slowly especially at the 90°F. storage, it is possible that sufficient free fatty acids might be liberated in time to cause rancidity in the concentrates stored at this temperature. The apparent absence of a cheesy flavor in concentrate 1 after 12 months and in the other concentrates after six months at 90°F. storage may be accounted for by the possible absence of bacterial and/or enzymatic activity.

The results of flavor evaluation of the butter concentrates, generally substantiated by those of the oxidation tests, indicate that with three evident exceptions detectable oxidized flavor development was prevented during the time of storage. The butter used in the manufacture of butter



concentrate 1 had been held for six months in cold storage prior to its use in the experiment. Apparently sufficient oxidation had occurred in the butter as indicated by the initial T.B.A. absorbance values to cause a tallowy flavor in butter concentrate 1a during its storage at 90°F. The presence of the added 4 p.p.m. diacetyl, possibly acting in a catalytic capacity, may have accounted for the tallowy flavor developing in butter concentrate 8 stored at both 70° and 90°F. Observations by Barnicoat (1937), in contradiction to this supposition, indicated that the addition of 4 to 6 p.p.m. to butter stored at 14°F. did not promote butterfat oxidation. However, the low temperature level used in this work might have minimized the catalytic effect of the diacetyl. Development of a tallowy flavor in butter concentrate 7 may have been masked by the strong flavor imparted to the two concentrates by the diacetyl.

It is evident that a caramelized flavor will develop with time in the butter concentrate, subsequent to heat treatment at 180°F. for 10 minutes, especially at a storage temperature of 90°F. The same phenomenon has been observed to occur in powdered milk if the temperature of the heat treatment during the processing is high. The flavor has been attributed to a reaction involving the lactose in the powder.



Data collected to date in this study of butter concentrates indicate that fat oxidation in the product can be controlled effectively at the temperature levels of 70° and 90°F. The development of cheesiness apparently also has been inhibited in the butter concentrates stored at 90°F. While the butterfat of the concentrate apparently will hydrolyze, the rate of reaction is much slower than in the modified butters and, therefore, if a rancid flavor does appear in the concentrates the time required for its development will likely be far longer than in the nitrogen packed butters. The evidence, therefore, indicates that the flavor stability of the butter concentrate stored at 90°F. may be superior to that of the nitrogen packed butters of the second modification stored at the same temperature.

Since this investigation is of an exploratory nature, the various experiments were inclined to be extensive rather than intensive. Extensive duplication of butters containing additives, such as preservatives and antioxidants, was not possible particularly in view of the fact that storage facilities were not available for incubation of replicate cans for the rather long storage time required in this investigation.

The technique employed for the emulsification and crystallization of the modified butter and butter concentrate in cans was only devised for research purposes and would be



quite unsuitable for commercial exploitation. For commercial production a continuous heat exchanger of the Votator type operated in the absence of air and having facilities for sealing the filled containers under an atmosphere of nitrogen would have to be developed.

#### CONCLUSIONS

1. First grade creamery butter packed in cans and held at 0°F., 45°F., room temperature and 95°F. did not have sufficient flavor stability at room temperature and 95°F. to warrant storage at these temperatures. In general, flavor deterioration occurred within the first month of storage at room temperature. Although cheesy and bitter flavors did become evident, the predominant off flavor to develop was rancidity.
2. Heat treatment of butter in sealed cans at 170° to 180°F. for 10 minutes, followed by re-emulsification of the serum and butterfat, materially reduced the number of bacteria in the butter. Little or no growth of the surviving organisms occurred at room temperature storage, subsequent to the heat treatment. In addition the development of the bacterially induced off flavors such as rancidity generally was prevented.



3. When bacterial activity was checked, a tallowy flavor caused by butterfat oxidation developed in the non-nitrogen packed modified butters stored at room temperature. Incorporation of antioxidants into these butters did not retard the development of the tallowy flavor.
4. A heat treatment of 170° to 180°F. for 10 minutes did not prevent the activity of bacteria in the modified butters which became severely oiled off at 90° or 95°F. A cheesy flavor developed rapidly in these butters.
5. Butterfat oxidation was inhibited in the nitrogen packed modified butters. Storage of these butters for at least 19 months at 70°F. without serious flavor deterioration indicated that this method of processing had potentialities for commercial exploitation.
6. Nitrogen packed modified butters stored at 90°F. developed a cheesy flavor after five to seven months of storage and a rancid flavor after 11 to 15 months of storage, but remained free of oxidative defects.
7. The prevention of oiling off and serum separation in the butters of the second modification stored at 90°F., by the incorporation of hardened oil, supplemented the action of the heat treatment and prevented serious bacterial growth.
8. With few exceptions the development of a tallowy flavor as well as a rancid or cheesy flavor was inhibited in the butter concentrate at a storage temperature as high as 90°F. for the time it was under observation. One



butter concentrate has been stored for 12 months while the others have been in storage for six months. There is some evidence that diacetyl added to the butter concentrate promoted fat oxidation.

9. The data available to date in this investigation indicates that butter concentrate may be a more suitable product than nitrogen packed butter especially for use at storage temperatures as high as 90°F.



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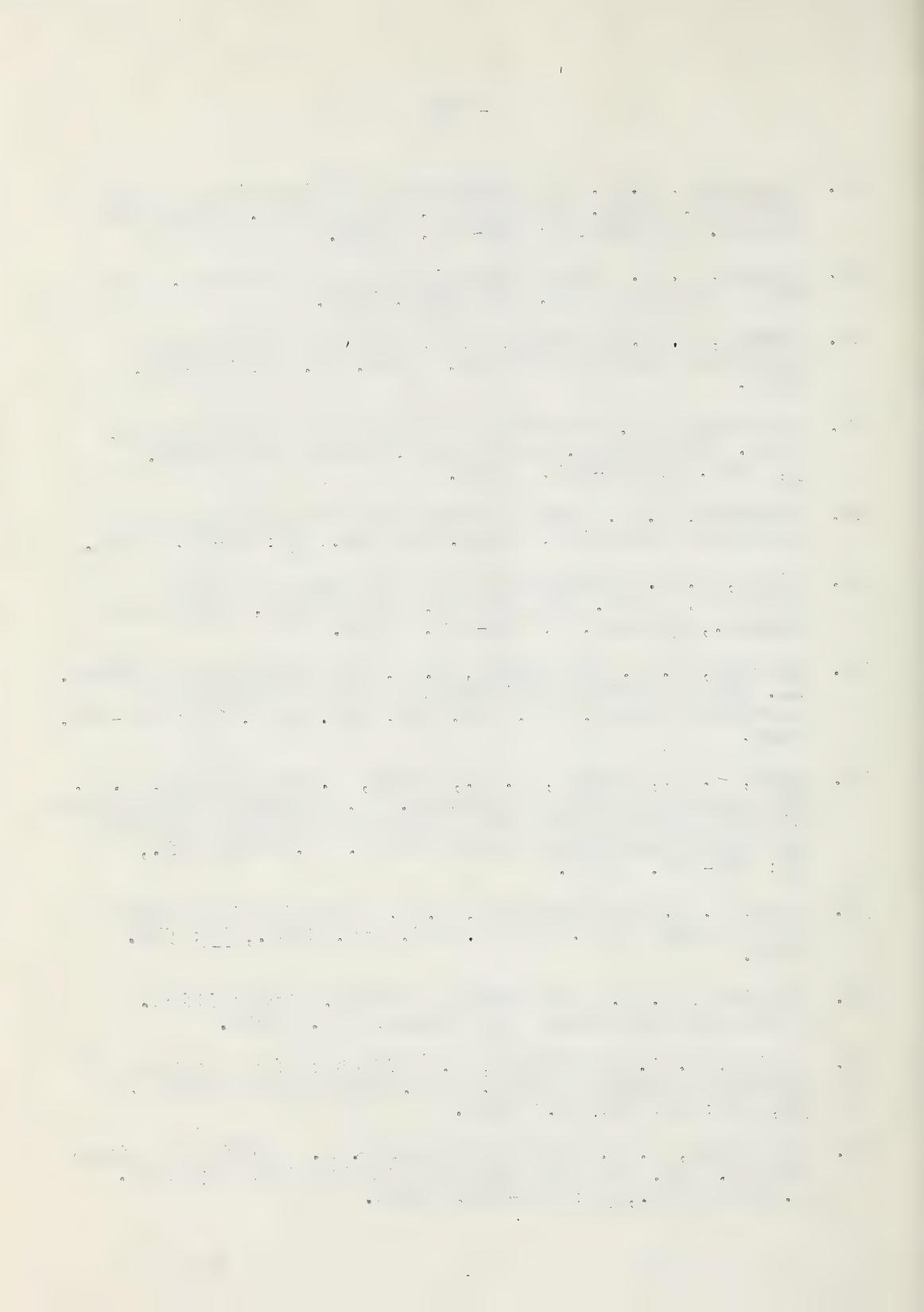
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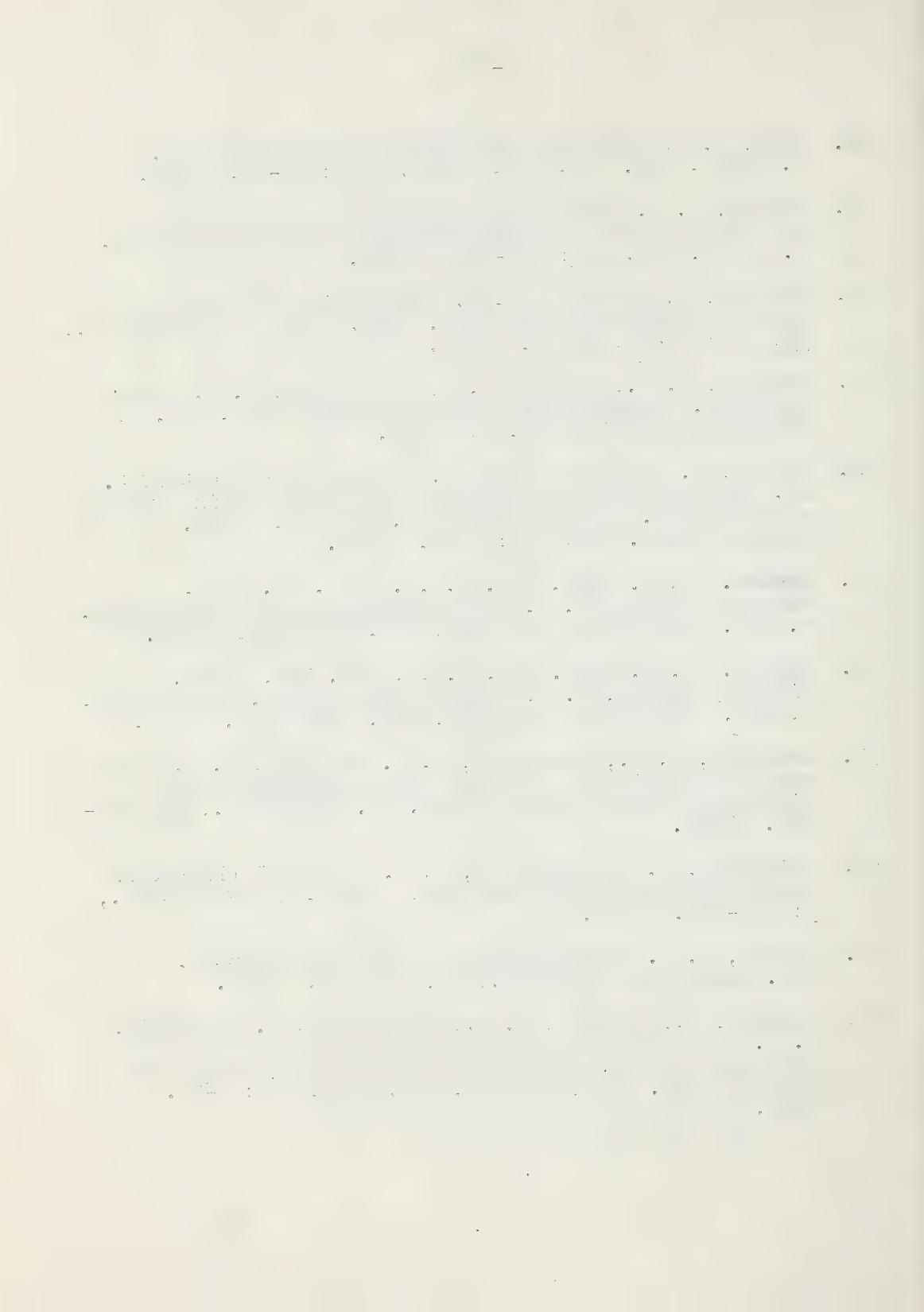
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TABLES AND FIGURES



TABLE 1. THE COMPOSITION OF THE BUTTERS  
OF THE FIRST MODIFICATION

Butter No.	Source of Butter	Concentration of incorporated additives expressed as percentage on a weight basis			
		Salt (sodium chloride)	Sodium benzoate	Tenox II	N.D.G.A.
8a	Laboratory	2	0	0	0
8b	"	0	0	0	0
9a	"	2	0	0.05	0
9b	"	0	0	0.05	0
10	"	2	0.1	0.05	0
11a	"	2	0.1	0	0
11b	"	0	0.1	0	0
16a	"	2	0.1	0	0.1
16b	"	0	0.1	0	0.1
12a	Creamery	2	0	0	0
12b	"	0	0	0	0
13a	"	2	0	0.05	0
13b	"	0	0	0.05	0
14	"	2	0.1	0.05	0
15a	"	2	0.1	0	0
15b	"	0	0.1	0	0



TABLE 2. THE COMPOSITION OF THE BUTTERS OF THE SECOND MODIFICATION

Butter No.	Source of Butter	Nitrogen replacement of the air	Conc. of incorporated additives expressed as percentage on a weight basis					Hardened oil
			Salt (sodium chloride)	Sodium benzoate	Tenox II	N.D.G.A.		
18	Creamery	Yes	2	0	0	0	0	
19	"	Yes	2	0.1	0	0.005	0	
32	"	No	2	0	0	0	0	
33	"	Yes	2	0	0	0	0	
34	"	No	2	0.1	0	0	0	
35	"	Yes	2	0.1	0	0	0	
36	"	No	2	0	0.05	0	0	
37	"	Yes	2	0	0.05	0	0	
38	"	No	2	0	0	0.005	0	
39	"	Yes	2	0	0	0.005	0	
40	"	No	2	0	0	0	5	
41	"	Yes	2	0	0	0	5	
42	"	No	2	0.1	0	0	5	
43	"	Yes	2	0.1	0	0	5	
44	"	No	2	0	0.05	0	5	
45	"	Yes	2	0	0.05	0	5	
46	"	No	2	0	0	0.005	5	
47	"	Yes	2	0	0	0.005	5	
52	Laboratory	No	2	0	0	0	0	
53	"	Yes	2	0	0	0	0	
54	"	No	2	0.1	0	0	0	
55	"	Yes	2	0.1	0	0	0	
56	"	No	2	0	0.05	0	0	
57	"	Yes	2	0	0.05	0	0	
58	"	No	2	0	0	0.005	0	
59	"	Yes	2	0	0	0.005	0	
60	"	No	2	0	0	0	5	
61	"	Yes	2	0	0	0	5	
62	"	No	2	0.1	0	0	5	
63	"	Yes	2	0.1	0	0	5	
64	"	No	2	0	0.05	0	5	
65	"	Yes	2	0	0.05	0	5	
66	"	No	2	0	0	0.005	5	
67	"	Yes	2	0	0	0.005	5	



TABLE 3. THE COMPOSITION OF THE BUTTER CONCENTRATE

Butter concentrate No.	Source of butterfat	Containing washed nitrogen	Water content	Conc. of incorporated additives expressed as percentage on a weight basis			
				Salt (sodium chloride)	Skim milk powder	N.D.G.A.	Diacetyl Hardened oil
1a	Creamery butter	No	0.20	2	4	0	0
1b	"	Yes	0.37	2	4	0	0
1c	"	No	0.43	2	4	0.005	0
1d	"	Yes	0.56	2	4	0.005	0
2a	"	No	--	1.25	4	0	0
2b	"	Yes	--	1.25	4	0	0
2c	"	No	--	1.25	4	0.005	0
2d	"	Yes	--	1.25	4	0.005	0
3a	"	No	0.24	1.25	4	0	0
3b	"	Yes	0.22	1.25	4	0	0
3c	"	No	0.16	1.25	4	0.005	0
3d	"	Yes	0.22	1.25	4	0.005	0
4a	Butter oil	No	0.24	1.25	4	0.005	0
4b	"	Yes	0.25	1.25	4	0	0
4c	"	No	0.25	1.25	4	0.005	0
4d	"	Yes	0.30	1.25	4	0.005	0
5	Creamery butter	No	0.27	1.25	4	0	0
6	"	Yes	--	1.25	4	0	0
7	"	No	--	1.25	4	0	4 ppm
8	"	Yes	--	1.25	4	0	4 ppm



TABLE 4. BACTERIAL PLATE COUNT OF BUTTERS  
PRELIMINARY INVESTIGATION

Butter No.	Initial Count	45° F.				Room (60°-80° F.)				95° F.			
		1 mo.	2 mo.	3 mo.	4 mo.	1 mo.	2 mo.	3 mo.	4 mo.	1 mo.	2 mo.	3 mo.	4 mo.
1	14.2	--	82	239	--	11,300	1500	170	--	9500	97	97	40
2	360	400	690	510	420	20,900	1310	450	--	6	0.4	<0.1	
3	29	79	97	78	210	3300	2580	129	--	430	<10	0.7	
4	12.5	4520	5200	3500	3600	9100	3200	1060	--	1270	260	40	
5	10	69	330	170	50	270	170	92	--	2900	75	390	
20	124	--	--	--	--	20	11	4.8	23	6	0.8	--	
21	18	--	--	--	--	480	75	32	10	11	270	--	
31	104	--	--	--	--	160	373	2	--	--	--	--	
51	0.05	--	--	--	--	11	10.2	2.4	--	--	--	--	



TABLE 5. FREE FAT ACIDITY VALUES OF BUTTERS  
PRELIMINARY INVESTIGATION

Butter No.	Free fat acidity value						Storage temperature and interval Room (60° ~ 80°F.)	95°F.
	45°F.			1 mo. 2 mo. 3 mo. 4 mo.				
	1 mo.	2 mo.	3 mo.	4 mo.	1 mo.	2 mo.	3 mo.	4 mo.
1	0.77	0.74	0.70	0.81	--	3.50	5.24	6.65
2	0.68	0.76	0.74	0.69	0.84	1.27	3.09	4.26
3	0.43	0.45	0.49	0.46	0.58	1.71	6.99	9.43
4	0.63	0.61	1.56	2.08	5.85	2.55	5.18	7.24
5	0.69	0.63	0.68	0.73	1.07	1.23	10.50	10.64
20	0.69	--	--	--	--	0.88	0.98	1.04
21	0.39	--	--	--	--	0.69	0.76	0.95
31	0.40	--	--	--	--	0.64	1.08	1.86
51	0.34	--	--	--	--	0.49	0.54	0.79



TABLE 6. IODOMETRIC PEROXIDE VALUES OF BUTTERS  
PRELIMINARY INVESTIGATION

Butter No.	Initial Value	Iodometric peroxide value						95°F.		
		Storage temperature and interval			Room (60°-80°F.)			95°F.		
		45°F.	1 mo. 2 mo. 3 mo. 4 mo.	1 mo. 2 mo. 3 mo. 4 mo.	1 mo. 2 mo. 3 mo. 4 mo.	1 mo. 2 mo. 3 mo. 4 mo.	1 mo. 2 mo. 3 mo. 4 mo.	1 mo. 2 mo. 3 mo. 4 mo.	1 mo. 2 mo. 3 mo. 4 mo.	1 mo. 2 mo. 3 mo. 4 mo.
1	0	0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0
2	0	0	0 0.62	0 0.50	0 0	0 0	0 0	0.86	0 0	0 0
3	0	0	0.46 1.24	1.06	0.88	1.02	1.82	—	0.64	0.34 0
4	0	0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0.78
5	0	0	0 0	0 0	0 0	0 0	0 0	0.42	0 0	—
20	0	—	—	—	0.38	0.90	1.14	1.24	0 0	—
21	0	—	—	—	0 0	0 0	0 0	0 0	0 0	—
31	0	—	—	—	0 0	0 0	0 0	—	—	—
51	0	—	—	—	0 0	0 0	0 0	—	—	—



TABLE 7. BACTERIAL COUNTS OF BUTTERS  
FIRST MODIFICATION

Butter* No.	Additives	Initial count	45°F.				Storage temperature and interval				Room (60°-80°F.)			
			1 mo.		2 mo.		3 mo.		4 mo.		1 mo.		2 mo.	
			1 mo.	2 mo.	3 mo.	4 mo.	1 mo.	2 mo.	3 mo.	4 mo.	1 mo.	2 mo.	3 mo.	4 mo.
8a		<0.01	0.01	0.2	0.02	0.03	2100	1400	640	560				
8b		<0.01	0.01	>800	2900	<10	260	700	53800	1380				
9a		<0.01	0.01	0.2	0.03	<0.01	2260	330	340	215				
9b		<0.01	<0.01	0.03	0.07	400	1820	1200	670	1180				
10	Benzoate	<0.01	<0.01	0.26	0.07	0.06	<0.01	0.1	0.1	0.03	<0.01			
11a	"	<0.01	<0.02	<0.01	0.01	0.10	0.6	0.01	0.01	0.01	<0.01			
11b	"	<0.01	<0.01	0.02	0.01	0.02	0.15	0.04	<0.01	<0.01	<0.01			
16a	"	<0.01	0.07	<0.01	<0.01	--	0.3	0.1	<0.01	<0.01	--			
16b	"	<0.01	0.04	0.02	<0.01	--	0.04	<0.01	<0.01	<0.01	<0.01			
12a		7	1.9	--	1.03	0.95	1.9	--	--	--	0.3	0.12		
12b		1.7	1.8	--	0.06	0.65	2.6	--	--	--	1.9	1.8		
13a		1.6	1.05	0.76	0.82	0.53	0.24	0.11	0.11	0.11	5.7	<1		
13b	Benzoate	0.77	0.16	0.07	0.17	0.17	15.5	3.2	8.9	8.9	0.38			
14	"	4.8	2.8	2.4	2.1	1.9	0.1	0.1	0.6	1.5	0.6	1.5		
15a	"	4.3	2.4	3.2	1.3	1.8	4	0.1	3.2	3.2	<1			
15b	"	6.9	4.7	6	13	5.2	2830	3580	1440	221				

\*Butters designated as "b" - unsalted; all other butters - salted.

5 2 7 8 4 5 9 0

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80 81 82 83 84 85 86 87 88 89 90 91 92 93 94 95 96 97 98 99 100

0 2 0 2 . 0 4 0 2 0 2 0 6

2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31

TABLE 8. FREE FAT ACIDITY VALUES OF BUTTERS  
FIRST MODIFICATION

Butter* No.	Initial value	Free fat acidity value							
		Storage temperature and interval			Room (60°-80°F.)				
		1 mo.	2 mo.	3 mo.	4 mo.	1 mo.	2 mo.		
8a	0.39	0.41	0.39	0.40	0.38	0.68	0.72	0.78	0.76
8b	0.39	0.79	1.51	0.51	1.55	2.53	1.23	2.86	3.96
9a	0.54	0.39	0.28	0.27	0.36	0.44	0.45	0.48	0.51
9b	0.54	0.43	0.38	0.39	0.55	0.49	0.61	0.47	0.50
10	0.41	0.42	0.44	0.43	0.44	0.46	0.46	0.46	0.51
11a	0.37	0.40	0.37	0.38	--	0.44	0.42	0.43	--
11b	0.37	0.41	0.38	0.36	--	0.42	0.40	0.41	--
16a	--	--	--	--	--	--	--	--	--
16b	--	--	--	--	--	--	--	--	--
12a	0.36	0.35	0.35	0.33	--	0.35	0.36	0.37	--
12b	0.36	0.37	0.40	0.33	--	0.39	0.46	0.47	--
13a	0.44	0.40	0.36	0.39	--	0.45	0.41	0.40	--
13b	0.44	0.43	0.40	0.45	--	0.47	0.46	0.52	--
14	0.46	0.43	0.38	0.42	--	0.43	0.44	0.52	--
15a	0.48	0.50	0.48	0.43	--	0.53	0.58	0.59	--
15b	0.48	0.49	0.53	0.49	--	0.62	0.70	0.75	--

\*Butters designated as "b" - unsalted; all other butters - salted.



TABLE 9. IODOMETRIC PEROXIDE VALUES OF BUTTERS  
FIRST MODIFICATION

Butter* No.	Initial value	Iodometric peroxide value							
		Storage temperature and interval				Room (60°-80°F.)			
		45°F.		1 mo. 2 mo. 3 mo. 4 mo.		1 mo. 2 mo. 3 mo. 4 mo.		1 mo. 2 mo. 3 mo. 4 mo.	
8a	0	0	0	0	0.42	0	0	0	0
8b	0	0	0	0	0	0	0	0	0
9a	0	0	0	0	0	0	0	0.44	0.80
9b	0	0	0	0	0	0	0	0	0.64
10	0	0	0	0	0	0	0	0	0.88
11a	0	0	0	0	--	0	0	0	--
11b	0	0	0	0	--	0	0	0	--
16a	0	0	0	0	--	0	0	0	--
16b	0	0	0	0	--	0	0	0	--
12a	0	0	0	0	--	0	0	0.42	--
12b	0	0	0	0	--	0	0	0.34	--
13a	0	0	0	0	--	0	0	1.02	0.36
13b	0	0	0	0	--	0	0	1.00	0.66
14	0	0	0	0	--	0	0	0.82	0.74
15a	0	0	0	0	--	0	0.86	0.43	--
15b	0	0	0	0	--	0	0	0	--

\*Butters designated as "b" unsalted; all other butters - salted.



TABLE 10. FLAVOR DEFECTS OF BUTTERS  
FIRST MODIFICATION

Butter* No.	Additives	Flavor defects**							
		Storage temperature and interval			Room (60°-80°F.)				
45°F.		1 mo.	2 mo.	3 mo.	4 mo.	1 mo.	2 mo.	3 mo.	4 mo.
8a		0	0	T		C	C	C	B
8b		0	B	R	B	S1.	S1.	S1.	B
9a		0	0	S1.T		0	T	T	T
9b		0	0	0	0	S1.T	S1.T	S1.T	T
10	"	0	0	0	0	S1.T	S1.T	S1.T	T
11a		0	0	0	0	S1.T	S1.T	S1.T	T
11b		0	0	0	0	S1.T	S1.T	S1.T	T
16a	N.D.G.A.	0	0	0	0	--	0	S1.T	T
16b	"	0	0	0	0	--	0	S1.T	T
12a		0	0	0	0	0	S1.T	S1.T	T
12b		0	0	0	0	0	S1.T	S1.T	T
13a	Tenox II	0	0	0	0	0	S1.T	S1.T	T
13b	"	0	0	0	0	0	S1.T	S1.T	T
14		0	0	0	0	0	S1.T	S1.T	T
15a		0	0	0	0	0	0	S1.T	T
15b		0	0	0	0	0	0	0	T

\*Butters designated as "b" - unsalted; all other butters - salted.

\*\*0 - No flavor defect

C - Cheesy

S1. - Slight

B - Bitter  
R - Rancid  
T - Tallowy



TABLE 11. BACTERIAL COUNTS OF CREAMERY BUTTERS  
SECOND MODIFICATION

Butter* No.	Additives	Initial count	Storage interval in months						Plate count		
			1	2	3	5	7	9	11	19	
32	None	2200	1600	17,200	1550	1400	--	--	--	--	
33	None	2600	5200	3000	7700	5950	730	435	825	480	
34	Benzoate	2000	5000	3300	1450	1450	--	--	--	--	
35	Benzoate	2700	3800	2800	1350	500	505	360	355	415	
36	Tenox II	3200	870	900	850	1000	--	--	--	--	
37	Tenox II	4300	2100	1200	1150	405	395	430	680	430	
38	N.D.G.A.	2200	800	500	1600	325	390	245	--	--	
39	N.D.G.A.	3100	1200	2350	4850	355	290	460	310	185	
40	None	2600	1600	1650	1000	1000	--	--	--	--	
41	None	3400	1250	1450	950	700	825	675	80	75	
42	Benzoate	2000	650	650	600	260	--	--	--	--	
43	Benzoate	3900	1000	350	450	550	350	245	115	85	
44	Tenox II	2100	750	900	800	700	--	--	--	--	
45	Tenox II	2900	250	700	900	650	1010	240	65	60	
46	N.D.G.A.	2400	400	950	315	200	--	--	--	--	
47	N.D.G.A.	3200	250	450	425	450	400	180	25	25	

\*Even numbered - non-nitrogen packed controls; odd numbered - nitrogen packed.



TABLE 12. BACTERIAL COUNTS OF LABORATORY BUTTERS  
SECOND MODIFICATION

Butter* No.	Additives	Initial count	Plate count						Storage interval in months	Stored at 70°F.	Storage interval in months	Stored at 90°F.
			1	2	3	5	7	9				
52	None	10	25	45	665	5	--	--	--	--	--	--
53	None	10	<100	240	170	<10	--	--	--	--	--	--
54	Benzoate	15	10	5	5	10	--	--	--	--	--	--
55	Benzoate	10	10	0	50	5	5	10	5	0	--	--
56	Tenox II	0	0	75	90	610	--	--	--	--	--	--
57	Tenox II	10	25	230	\$15	55	5	0	0	5	5	5
58	N.D.G.A.	10	110	115	170	30	25	40	35	--	--	--
59	N.D.G.A.	10	30	115	75	940	60	25	5	5	5	5
60	None	10	20	820	95	5	--	--	--	--	--	--
61	None	10	60	600	150	20	--	--	--	--	--	--
62	Benzoate	10	0	5	10	15	--	--	--	--	--	--
63	Benzoate	10	0	10	15	20	<10	0	0	0	45	--
64	Tenox II	10	340	715	1140	60	--	--	--	--	--	--
65	Tenox II	10	115	140	0	5	10	30	5	5	20	--
66	N.D.G.A.	15	55	15	700	--	--	--	--	--	--	--
67	N.D.G.A.	--	1650	345	440	<10	5	5	5	35	5	575

\*Even numbered - non-nitrogen packed controls; odd numbered - nitrogen packed.

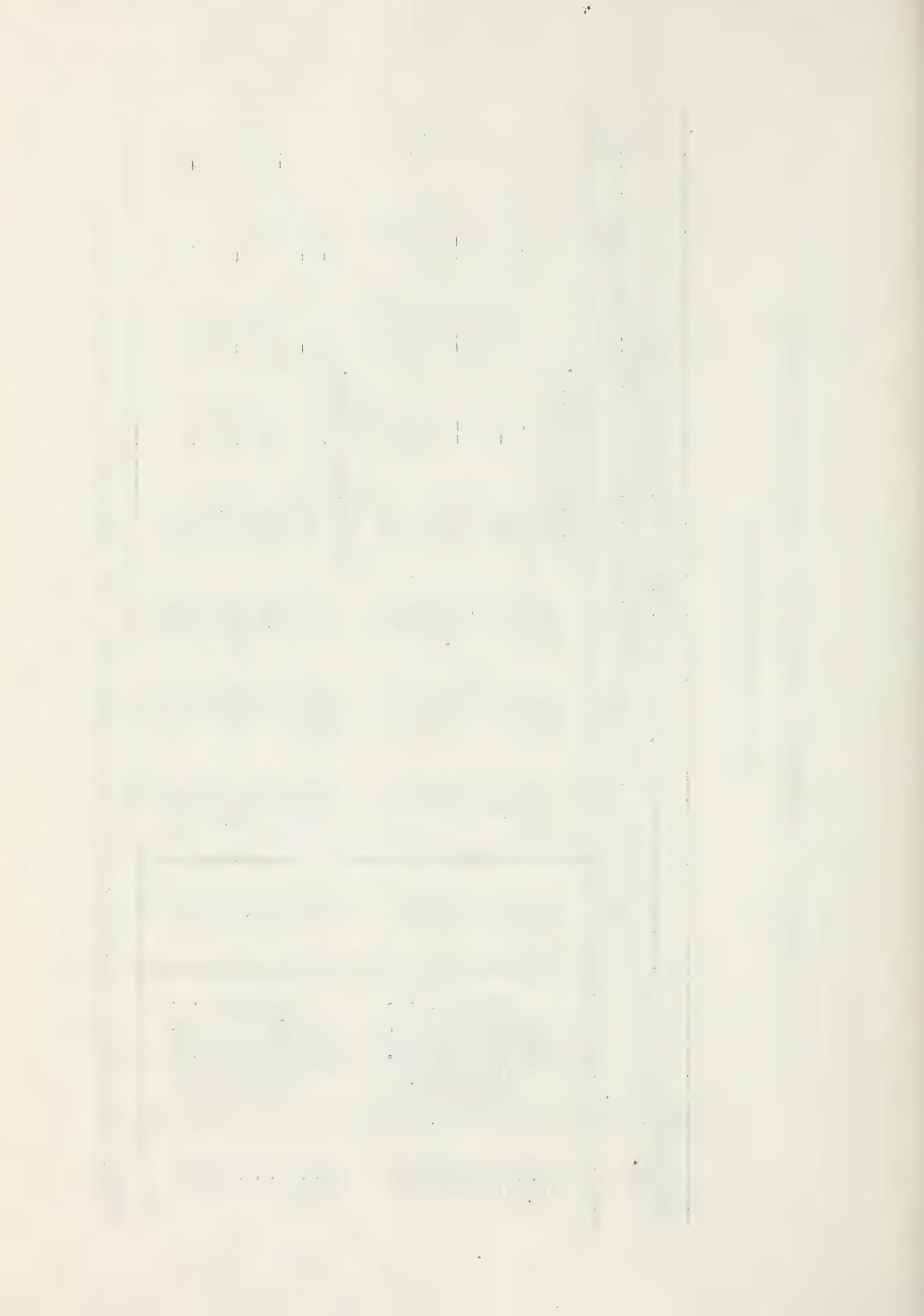


TABLE 13. FREE FAT ACIDITY VALUES OF CREAMERY BUTTERS  
SECOND MODIFICATION

Butter* No.	Additives	Initial value	Free fat acidity value						Storage interval in months	Stored at 70°F.	Stored at 90°F.
			1	2	3	5	7	9			
32	None	0.41	0.49	0.48	0.47	0.55	0.55	0.59	0.63	0.70	0.69
33	None	0.44	0.43	0.47	0.46	0.52	0.55	0.59	0.63	0.70	0.75
34	Benzoate	0.40	0.44	0.48	0.45	0.54	0.53	0.57	0.66	0.72	0.75
35	Benzoate	0.39	0.42	0.46	0.45	0.49	0.53	0.57	0.66	0.72	0.78
36	Tenox II	0.41	0.47	0.47	0.47	0.50	0.55	0.57	0.66	0.72	0.75
37	Tenox II	0.40	0.45	0.47	0.52	0.55	0.56	0.62	0.66	0.74	0.75
38	N.D.G.A.	0.42	0.44	0.48	0.57	0.54	0.62	0.66	0.74	0.75	0.79
39	N.D.G.A.	0.40	0.44	0.49	0.53	0.54	0.54	0.58	0.67	0.74	0.75
40	None	0.44	0.45	0.56	0.61	0.78	0.78	0.90	1.00	1.24	1.26
41	None	0.40	0.53	0.58	0.60	0.77	0.77	0.90	1.00	1.24	1.37
42	Benzoate	0.38	0.49	0.59	0.64	0.86	0.86	0.97	1.09	1.33	1.84
43	Benzoate	0.43	0.49	0.52	0.63	0.80	0.80	0.97	1.09	1.33	1.68
44	Tenox II	0.40	0.51	0.60	0.62	0.78	0.78	0.97	1.09	1.33	1.97
45	Tenox II	0.42	0.49	0.57	0.59	0.76	0.76	0.93	1.11	1.32	1.34
46	N.D.G.A.	0.43	0.55	0.60	0.63	0.83	0.83	0.93	1.11	1.32	1.67
47	N.D.G.A.	0.42	0.52	0.58	0.68	0.75	0.75	0.93	1.07	1.26	1.40

\*Even numbered - non-nitrogen packed controls; odd numbered - nitrogen packed.



TABLE 14. FREE FAT ACIDITY VALUES OF LABORATORY BUTTERS  
SECOND MODIFICATION

Butter* No.	Additives	Initial value	Free fat acidity value								
			1	2	3	5	7	9	11	13	15
Storage interval in months											
			Stored at 70°F.								
52	None	0.37	0.36	0.38	0.38	0.50	--	--	--	--	--
53	None	0.36	0.37	0.37	0.39	0.46	--	--	--	--	--
54	Benzoate	0.40	0.42	0.38	0.43	0.55	--	--	--	--	--
55	Benzoate	0.42	0.42	0.40	0.45	0.54	0.56	0.59	0.58	0.65	0.71
56	Tenox II	0.43	0.35	0.35	0.39	0.42	--	--	--	--	0.54
57	Tenox II	0.38	0.36	0.35	0.37	0.41	0.44	0.44	0.46	0.51	0.50
58	N.D.G.A.	0.35	0.37	0.37	0.38	0.43	0.47	0.45	0.48	0.51	0.51
59	N.D.G.A.	0.37	0.35	0.36	0.39	0.43	0.48	0.45	0.48	0.50	0.51
			Stored at 90°F.								
60	None	0.43	0.50	0.48	0.54	0.63	--	--	--	--	--
61	None	0.44	0.48	0.49	0.55	0.64	--	--	--	--	--
62	Benzoate	0.45	0.51	0.52	0.62	0.80	--	--	--	--	--
63	Benzoate	0.44	0.55	0.56	0.64	0.81	0.93	1.05	1.28	1.34	1.70
64	Tenox II	0.42	0.45	0.51	0.55	0.70	--	--	--	--	1.86
65	Tenox II	0.44	0.45	0.49	0.53	0.65	0.70	0.84	0.96	1.01	1.11
66	N.D.G.A.	0.45	0.42	0.46	0.51	0.63	--	--	--	--	1.35
67	N.D.C.A.	0.42	0.44	0.46	0.50	0.60	0.67	0.80	0.90	0.91	1.10
			19								

\*Even numbered - non-nitrogen packed controls; odd numbered - nitrogen packed.

2      3      4      5      6      7      8      9      10      11      12      13      14      15      16      17      18      19      20      21      22      23      24      25      26      27      28      29      30      31      32      33      34      35      36      37      38      39      40      41      42      43      44      45      46      47      48      49      50      51      52      53      54      55      56      57      58      59      60      61      62      63      64      65      66      67      68      69      70      71      72      73      74      75      76      77      78      79      80      81      82      83      84      85      86      87      88      89      90      91      92      93      94      95      96      97      98      99      100

10. *What is the best way to increase the number of people who use a particular service?*

TABLE 15. IODOMETRIC PEROXIDE VALUES OF CREAMERY BUTTERS  
SECOND MODIFICATION

Butter* No.	Additives	Initial value	Iodometric peroxide value							
			1	2	3	5	7	9	11	13
32	None	0	0	0	0.84	1.28	-	-	-	-
33	None	0	0	0	0	0	0	0	0	0
34	Benzoate	0	0	1.08	1.86	-	-	-	-	-
35	Benzoate	0	0	0	0	0	0	0	0	0
36	Tenox II	0	0	0.98	1.48	-	-	-	-	-
37	Tenox II	0	0	0	0	0	0	0	0	0
38	N.D.G.A.	0	0	0	0	0	0	0	0	0
39	N.D.G.A.	0	0	0	0	0	0	0	0	0
40	None	0	1.30	1.46	1.06	0	-	-	-	-
41	None	0	0	0	0	0	0	0	0	0
42	Benzoate	0	0.98	1.08	0.82	0	-	-	-	-
43	Benzoate	0	0	0	0	0	0	0	0	0
44	Tenox II	0	1.04	0	1.24	0	-	-	-	-
45	Tenox II	0	0	0	0	0	0	0	0	0
46	N.D.G.A.	0	0	0	0.60	0	-	-	-	-
47	N.D.G.A.	0	0	0	0	0	0	0	0	0

\*Even numbered - non-nitrogen packed controls; odd numbered - nitrogen packed.



TABLE 16. IODOMETRIC PEROXIDE VALUES OF LABORATORY BUTTERS  
SECOND MODIFICATION

Butter* No.	Additives	Initial value	Iodometric peroxide value								
			Storage interval in months								
			1	2	3	5	7	9	11	13	15
Stored at 70°F.											
52	None	0	0	0	0.78	0	-	-	-	-	-
53	None	0	0	0	0	-	-	-	-	-	-
54	Benzoate	0	0	0.66	1.18	1.28	-	-	-	-	-
55	Benzoate	0	0	0	0	0.36	0	0.60	0	0.56	0
56	Tenox II	0	0	0	0.72	0.76	-	-	-	-	-
57	Tenox II	0	0	0	0	0	0	0	0	0.36	0
58	N.D.G.A.	0	0	0	0	0	0	1.20	0.42	-	-
59	N.D.G.A.	0	0	0	0	0	0	0	0	0.32	0
Stored at 90°F.											
60	None	0	0	0	0.88	0.70	-	-	-	-	-
61	None	0	0	0	0.52	0	-	-	-	-	-
62	Benzoate	0	0	0	1.30	0.62	-	-	-	-	-
63	Benzoate	0	0	0	0	0	0	0	0	0	0
64	Tenox II	0	0	0.98	1.16	0	-	-	-	-	-
65	Tenox II	0	0	0	0	0	0	0	0	0	0
66	N.D.G.A.	0	0	0	0.34	0	-	-	-	-	-
67	N.D.G.A.	0	0	0	0	0	0	0	0	0	0

\*Even numbered - non-nitrogen packed controls; odd numbered - nitrogen packed.



TABLE 17. T.B.A. ABSORBANCE VALUES OF CREAMERY BUTTERS  
SECOND MODIFICATION

Butter* No.	Additives	Initial value	T.B.A. absorbance value								
			Storage interval in months								
			1	2	3	5	7	9	11	13	15
Stored at 70°F.											
32	None	0.030	0.119	0.118	0.167	0.235	—	—	—	—	—
33	None	0.031	0.085	0.112	0.087	0.094	0.036	0.031	0.022	0.025	0.015
34	Benzoate	0.028	0.171	0.222	0.158	0.233	—	—	—	—	—
35	Benzoate	0.057	0.057	0.073	0.078	0.059	0.052	0.036	0.026	0.022	0.017
36	Tenox II	0.040	0.113	0.125	0.182	0.225	—	—	—	—	—
37	Tenox II	0.039	0.077	0.072	0.076	0.064	0.040	0.035	0.019	0.025	0.012
38	N.D.G.A.	0.040	0.050	0.046	0.071	0.097	0.093	0.091	—	—	—
39	N.D.G.A.	0.029	0.048	0.030	0.054	0.035	0.033	0.037	0.025	0.022	0.012
Stored at 90°F.											
40	None	0.034	0.209	0.185	0.089	0.040	—	—	—	—	—
41	None	0.034	0.077	0.034	0.041	0.028	0.034	0.039	0.026	0.025	0.020
42	Benzoate	0.043	0.180	0.220	0.098	0.046	—	—	—	—	0.017
43	Benzocate	0.041	0.036	0.042	0.041	0.026	0.033	0.041	0.033	0.029	0.019
44	Tenox II	0.036	0.169	0.087	0.140	0.046	—	—	—	—	0.017
45	Tenox II	0.042	0.062	0.094	0.043	0.034	0.036	0.048	0.028	0.027	0.017
46	N.D.G.A.	0.041	0.062	0.089	0.123	0.045	—	—	—	—	0.015
47	N.D.G.A.	0.039	0.050	0.043	0.055	0.027	0.034	0.031	0.031	0.018	0.020

\*Even numbered - non-nitrogen packed controls; odd numbered - nitrogen packed.



TABLE 18. T.B.A. ABSORBANCE VALUES OF LABORATORY BUTTERS  
SECOND MODIFICATION

Butter* No.	Additives	Initial value	T.B.A. absorbance value									
			Storage interval in months			Stored at 70°F.						
			1	2	3	5	7	9	11	13	15	19
52	None	0.041	0.062	0.086	0.184	0.071	--	--	--	--	--	--
53	None	0.050	0.060	0.071	0.121	0.069	--	--	--	--	--	--
54	Benzoate	0.049	0.076	0.254	0.233	0.280	--	--	--	--	--	--
55	Benzoate	0.043	0.022	0.067	0.093	0.094	0.095	0.141	0.143	0.091	0.034	0.029
56	Tenox II	0.053	0.080	0.090	0.119	0.153	--	--	--	--	--	--
57	Tenox II	0.054	0.059	0.063	0.089	0.077	0.072	0.060	0.042	0.034	0.028	0.029
58	N.D.G.A.	0.039	0.055	0.063	0.067	0.064	0.115	0.202	0.132	--	--	--
59	N.D.C.A.	0.020	0.049	0.047	0.043	0.042	0.041	0.052	0.035	0.022	0.012	0.020
60	None	0.053	0.132	0.184	0.135	0.087	--	--	--	--	--	--
61	None	0.060	0.153	0.048	0.059	0.027	--	--	--	--	--	--
62	Benzoate	0.053	0.171	0.215	0.274	0.144	--	--	--	--	--	--
63	Benzoate	0.050	0.074	0.036	0.031	0.034	0.040	0.026	0.031	0.041	0.018	0.021
64	Tenox II	0.041	0.201	0.216	0.215	0.046	--	--	--	--	--	--
65	Tenox II	0.041	0.082	0.078	0.029	0.025	0.027	0.030	0.026	0.031	0.015	0.019
66	N.D.G.A.	0.059	0.080	0.089	0.082	0.060	--	--	--	--	--	--
67	N.D.C.A.	0.055	0.083	0.041	0.036	0.030	0.031	0.025	0.035	0.026	0.020	0.022

\*Even numbered - non-nitrogen packed controls; odd numbered - nitrogen packed.

0 5 2 8 3 6 7 0 9 8 6 9 7 8 9

9 7 5 4 3 2 0 8 3 7 5 6 4 6 8

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TABLE 19. FLAVOR DEFECTS IN CREAMERY BUTTERS

## SECOND MODIFICATION

Butter* No.	Additives	Storage interval in months									Flavor defects**		
		1	2	3	5	7	9	11	13	15	19	Stored at 70° F.	Stored at 90° F.
32	None	0	0	T	T	T	T	T	T	T	T	SI.T	SI.T
33	None	0	0	0	0	0	0	0	0	0	0	SI.T	SI.T
34	Benzocate	0	0	T	T	T	T	T	T	T	T	SI.T	SI.T
35	Benzocate	0	0	0	0	0	0	0	0	0	0	SI.F	SI.F
36	Tenox II	0	0	SI.T	SI.F	SI.F							
37	Tenox II	0	0	0	0	0	0	0	0	0	0	SI.F	SI.F
38	N.D.G.A.	0	0	0	0	0	0	0	0	0	0	SI.F	SI.F
39	N.D.G.A.	0	0	0	0	0	0	0	0	0	0	SI.F	SI.F
40	None	0	0	T	T	T	T	T	T	T	T	SI.C	SI.C
41	None	0	0	0	0	0	0	0	0	0	0	SI.C	SI.C
42	Benzocate	0	0	T	T	T	T	T	T	T	T	SI.C	SI.C
43	Benzocate	0	0	0	0	0	0	0	0	0	0	SI.C	SI.C
44	Tenox II	0	0	T	T	T	T	T	T	T	T	SI.C	SI.C
45	Tenox II	0	0	0	0	0	0	0	0	0	0	SI.C	SI.C
46	N.D.G.A.	0	0	T	T	T	T	T	T	T	T	SI.C	SI.C
47	N.D.G.A.	0	0	0	0	0	0	0	0	0	0	SI.C	SI.C

\*Even numbered - non-nitrogen packed controls; odd numbered - nitrogen packed.

\*\*O - No flavor defect  
O - No cheesy  
M - Metallic  
T - TallowyC - Cheesy  
R - Rancid

F - Foreign

S1. - Slight



TABLE 20. FLAVOR DEFECTS IN LABORATORY BUTTERS  
SECOND MODIFICATION

Butter* No.	Additives	Storage interval in months										Flavor defects**	
		1	2	3	5	7	9	11	13	15	19	Stored at 70° F.	Stored at 90° F.
52	None	0	S1.T	T	T	T	T	T	T	T	T	--	--
53	None	0	S1.T	S1.T	T	T	T	T	T	T	T	--	--
54	Benzoate	0	T	T	T	T	T	T	T	T	T	--	--
55	Benzoate	0	0	0	0	0	0	0	0	0	0	S1.T	S1.T
56	Tenox II	0	0	S1.T	T	T	T	T	T	T	T	--	--
57	Tenox II	0	0	0	0	0	0	0	0	0	0	S1.T	S1.T
58	N.D.G.A.	0	0	0	0	0	0	0	0	0	0	T, M	S1.F.
59	N.D.G.A.	0	0	0	0	0	0	0	0	0	0	S1.F.	S1.F.
60	None	0	T	T	S1.T	S1.T	T	T	T	T	T	--	--
61	None	0	S1.T	T	T	T	T	T	T	T	T	--	--
62	Benzoate	0	0	0	S1.C	--	--						
63	Benzoate	0	0	0	T	T	T	T	T	T	T	S1.R	S1.R
64	Tenox II	0	T	T	T	T	T	T	T	T	T	--	--
65	Tenox II	0	0	0	0	0	S1.C	S1.C	S1.C	S1.C	S1.C	--	--
66	N.D.C.A.	0	M	M	M	M	T	T	T	T	T	S1.R	S1.R
67	N.D.G.A.	0	0	0	F	F	F	F	F	F	F	S1.C	S1.C

\*Even numbered - non-nitrogen packed controls; odd numbered - nitrogen packed.

\*\*O - No flavor defect  
M - Metallic  
T - Tallowy

C - Cheesy  
R - Rancid  
F - Foreign  
S1. - Slight



TABLE 21. FREE FAT ACIDITY VALUES OF BUTTER CONCENTRATES

Butter Concentrate No.	Initial value	Free fat acidity value					
		Storage interval in months at 70°F.					
		2	4	6	8	10	12
1a	0.45	0.45	0.46	0.47	0.47	0.48	0.48
1b	0.44	0.41	0.45	0.47	0.47	0.48	0.52
1c	0.45	0.40	0.46	0.47	0.46	0.50	0.52
1d	0.44	0.42	0.46	0.50	0.48	0.49	0.53
2a	0.49	0.56	0.57	0.57			
2b	0.50	0.56	0.55	0.55			
2c	0.52	0.56	0.53	0.57			
2d	0.51	0.55	0.57	0.56			
3a	0.47	0.42	0.44	0.44			
3b	0.47	0.43	0.45	0.44			
3c	0.47	0.42	0.45	0.41			
3d	0.46	0.43	0.44	0.41			
4a	0.47	0.44	0.45	0.48			
4b	0.47	0.45	0.47	0.46			
4c	0.48	0.45	0.48	0.50			
4d	0.48	0.44	0.48	0.50			
5	0.50	0.51	0.52	0.60			
6	0.45	0.44	0.44	0.50			
7	0.55	0.51	0.54	0.57			
8	0.49	0.48	0.50	0.46			



TABLE 22. FREE FAT ACIDITY VALUES OF BUTTER CONCENTRATES

Butter Concentrate No.	Initial value	Free fat acidity value					
		Storage interval in months at 90°F.					
		2	4	6	8	10	12
1a	0.45	0.51	0.52	0.53	0.54	0.52	0.62
1b	0.44	0.50	0.54	0.62	0.66	0.66	0.81
1c	0.45	0.48	0.56	0.61	0.71	0.70	0.83
1d	0.44	0.50	0.64	0.64	0.71	0.73	0.84
2a	0.49	0.56	0.58	0.59			
2b	0.50	0.55	0.56	0.58			
2c	0.52	0.57	0.58	0.58			
2d	0.51	0.55	0.58	0.58			
3a	0.47	0.43	0.47	0.48			
3b	0.47	0.44	0.45	0.49			
3c	0.47	0.43	0.46	0.50			
3d	0.46	0.43	0.47	0.46			
4a	0.47	0.46	0.49	0.53			
4b	0.47	0.46	0.50	0.54			
4c	0.48	0.48	0.51	0.59			
4d	0.48	0.48	0.51	0.60			
5	0.50	0.49	0.53	0.61			
6	0.45	0.47	0.50	0.55			
7	0.55	0.48	0.56	0.63			
8	0.49	0.48	0.50	0.55			



TABLE 23. IODOMETRIC PEROXIDE VALUES OF BUTTER CONCENTRATES

Butter Concentrate No.	Initial value	Iodometric peroxide value					
		Storage interval in months at 70°F.					
		2	4	6	8	10	12
1a	0	0	0	0	0	0	0
1b	0	0	0	0	0	0	0
1c	0	0	0	0	0	0	0
1d	0	0	0	0	0	0	0
2a	0	0	0	0			
2b	0	0	0	0			
2c	0	0	0	0			
2d	0	0	0	0			
3a	0	0	0	0			
3b	0	0	0	0			
3c	0	0	0	0			
3d	0	0	0	0			
4a	0	0	0	0			
4b	0	0	0	0			
4c	0	0	0	0			
4d	0	0	0	0			
5	0	0	0	0			
6	0	0	0	0			
7	0	0	0	0.32			
8	0	0.90	0.42	0.64			



TABLE 24. IODOMETRIC PEROXIDE VALUES OF BUTTER CONCENTRATE

Butter Concentrate No.	Initial value	Iodometric peroxide value					
		2	4	6	8	10	12
1a	0	0	0.70	0	0	0	0
1b	0	0	0	0	0	0	0
1c	0	0	0	0	0	0	0
1d	0	0	0	0	0	0	0
2a	0	0	0	0			
2b	0	0	0	0			
2c	0	0	0	0			
2d	0	0	0	0			
3a	0	0	0	0			
3b	0	0	0	0			
3c	0	0	0	0			
3d	0	0	0	0			
4a	0	0	0	0			
4b	0	0	0	0			
4c	0	0	0	0			
4d	0	0	0	0			
5	0	0	0	0			
6	0	0	0	0			
7	0	0.62	0	0.50			
8	0	0.78	0.56	0.66			



TABLE 25. FERRIC THIOCYANATE PEROXIDE VALUES OF  
BUTTER CONCENTRATES

Butter Concentrate No.	Initial value	Ferric thiocyanate peroxide value					
		Storage interval in months at 70°F.					
		2	4	6	8	10	12
1a	0.58	0.54	0.35	0.33	0.31	0.33	0.28
1b	--	0.52	0.56	0.24	0.21	0.19	0.13
1c	--	0.60	0.43	0.26	0.17	0.23	0.15
1d	0.52	0.58	0.36	0.28	0.19	0.19	0.12
2a	0.12	0.22	0.30	0.33			
2b	0.17	0.25	0.32	0.35			
2c	0.17	0.21	0.29	0.28			
2d	0.19	0.19	0.35	0.30			
3a	0.24	0.17	0.23	0.23			
3b	0.22	0.17	0.29	0.26			
3c	0.26	0.19	0.19	0.26			
3d	0.22	0.19	0.17	0.23			
4a	0.30	0.25	0.25	0.21			
4b	0.31	0.25	0.27	0.39			
4c	0.41	0.27	0.17	0.25			
4d	0.46	0.21	0.17	0.19			
5	0.24	0.25	0.21	0.21			
6	0.19	0.25	0.21	0.21			
7	0.21	0.28	0.42	0.52			
8	0.19	1.21	0.85	1.00			



TABLE 26. FERRIC THIOCYANATE PEROXIDE VALUES OF  
BUTTER CONCENTRATES

Butter Concentrate No.	Initial value	Ferric thiocyanate peroxide values					
		Storage interval in months at 90°F.					
		2	4	6	8	10	12
1a	0.58	1.00	0.52	0.39	0.45	0.27	0.31
1b	--	0.48	0.23	0.30	0.33	0.15	0.19
1c	--	0.43	0.30	0.26	0.33	0.17	0.26
1d	0.52	0.49	0.31	0.26	0.43	0.20	0.17
2a	0.12	0.27	0.32	0.35			
2b	0.17	0.38	0.37	0.31			
2c	0.17	0.22	0.32	0.26			
2d	0.19	0.21	0.18	0.30			
3a	0.24	0.21	0.30	0.21			
3b	0.22	0.25	0.29	0.30			
3c	0.26	0.25	0.30	0.20			
3d	0.22	0.19	0.41	0.37			
4a	0.30	0.42	0.35	0.28			
4b	0.31	0.48	0.39	0.31			
4c	0.42	0.38	0.35	0.26			
4d	0.46	0.33	0.32	0.35			
5	0.24	0.22	0.23	0.17			
6	0.19	0.18	0.50	0.26			
7	0.21	1.06	0.45	0.60			
8	0.19	1.06	0.93	0.94			



TABLE 27. T.B.A. ABSORBANCE VALUES OF BUTTER CONCENTRATES

Butter Concentrate No.	T.B.A. absorbance value						
	Initial value	Storage interval in months at 70°F.					
		2	4	6	8	10	12
1a	0.087	0.045	0.041	0.054	0.058	0.041	0.046
1b	0.094	0.053	0.050	0.046	0.046	0.039	0.048
1c	0.102	0.052	0.036	0.031	0.048	0.036	0.046
1d	0.090	0.058	0.041	0.046	0.041	0.041	0.046
2a	0.057	0.046	0.063	0.055			
2b	0.052	0.053	0.062	0.048			
2c	0.050	0.048	0.064	0.049			
2d	0.053	0.058	0.064	0.046			
3a	0.057	0.057	0.059	0.041			
3b	0.060	0.058	0.065	0.048			
3c	0.058	0.063	0.054	0.041			
3d	0.060	0.055	0.054	0.043			
4a	0.050	0.059	0.046	0.053			
4b	0.046	0.046	0.052	0.059			
4c	0.046	0.054	0.046	0.043			
4d	0.055	0.050	0.052	0.046			
5	0.062	0.062	0.048	0.048			
6	0.046	0.069	0.054	0.046			
7	0.046	0.052	0.053	0.053			
8	0.050	0.160	0.067	0.044			



TABLE 28. T.B.A. ABSORBANCE VALUES OF BUTTER CONCENTRATES

Butter Concentrate No.	T.B.A. absorbance value						
	Initial value	Storage interval in months at 90°F.					
		2	4	6	8	10	12
1a	0.087	0.055	0.057	0.081	0.048	0.046	0.041
1b	0.094	0.046	0.041	0.059	0.043	0.053	0.072
1c	0.102	0.049	0.041	0.054	0.046	0.047	0.077
1d	0.090	0.057	0.050	0.054	0.041	0.050	0.076
2a	0.057	0.080	0.076	0.057			
2b	0.052	0.089	0.073	0.050			
2c	0.050	0.046	0.073	0.043			
2d	0.053	0.055	0.069	0.046			
3a	0.057	0.062	0.072	0.047			
3b	0.060	0.073	0.068	0.060			
3c	0.058	0.062	0.063	0.048			
3d	0.060	0.060	0.064	0.042			
4a	0.050	0.048	0.052	0.036			
4b	0.046	0.080	0.071	0.039			
4c	0.046	0.063	0.062	0.041			
4d	0.055	0.067	0.063	0.048			
5	0.062	0.041	0.053	0.036			
6	0.046	0.048	0.054	0.065			
7	0.046	0.108	0.050	0.046			
8	0.050	0.098	0.072	0.060			



TABLE 29. ESTIMATION OF IRON AND COPPER CONCENTRATIONS IN BUTTERS  
OF SECOND MODIFICATION AND IN BUTTER CONCENTRATES

Sample	Storage interval and temperature prior to determination		Concentration of metal in p.p.m.	
	Interval-mo.	Temperature-°F.	Iron	Copper
Butter 37	17	0	0.52	0.055
" 37	17	70	0.58	0.070
" 45	17	0	0.52	0.040
" 45	17	90	0.54	0.040
" 57	17	0	1.48	0.025
" 57	17	70	1.44	0.039
" 65	17	0	1.41	0.040
" 65	17	90	1.60	0.040
Butter Concentrate 1	7	0	0.47	0.085
" 2	7	0	0.48	0.059
" 3	7	0	0.49	0.065
" 4	7	0	0.43	0.050
" 5	7	0	0.48	0.062
" 6	7	0	0.53	0.067
" 7	7	0	0.58	0.072
" 8	7	0	0.48	0.062



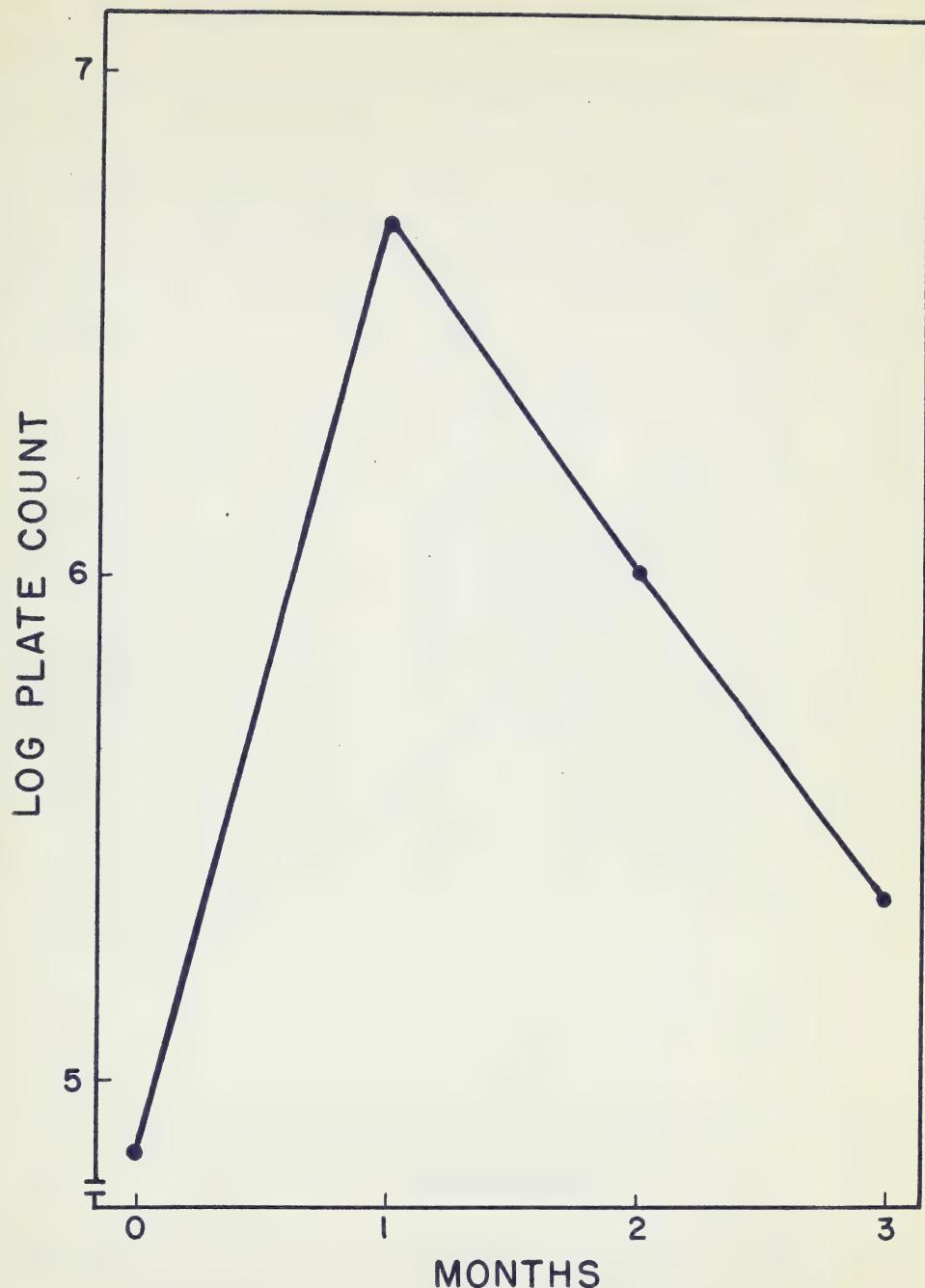


Figure 1. Trend of the average bacterial population in nine butters of the preliminary investigation stored at room temperature.



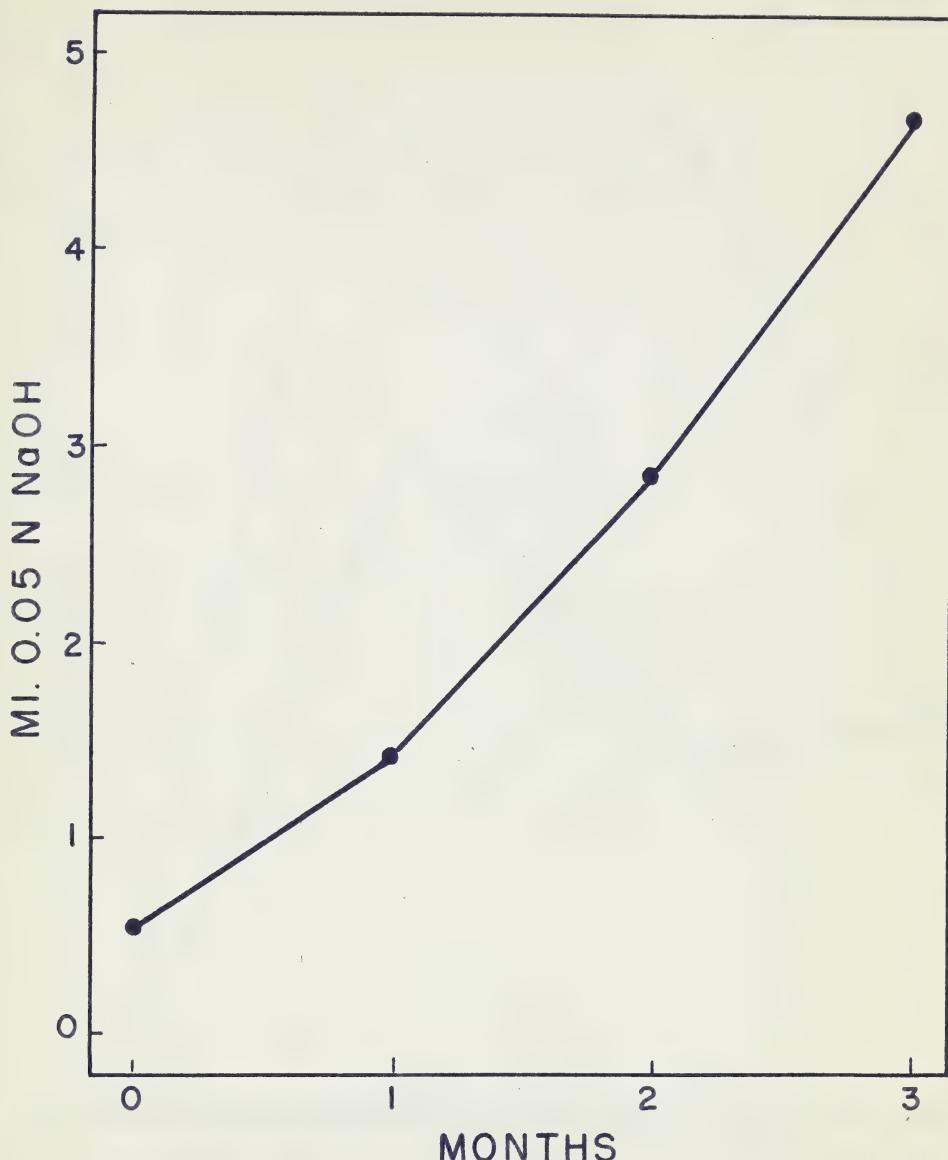


Figure 2. Increase in the average free fat acidity level of nine butters of the preliminary investigation stored at room temperature.



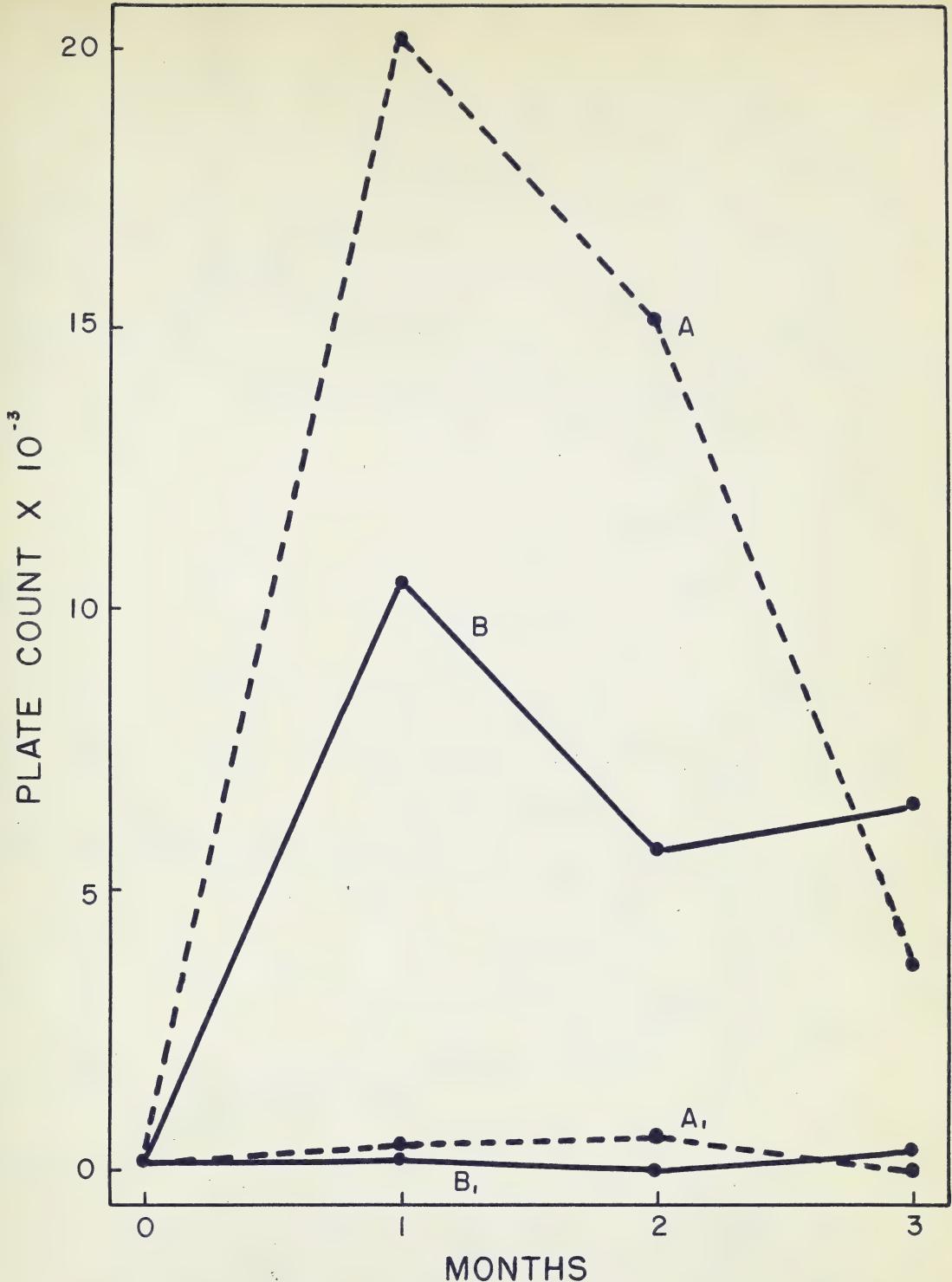


Figure 3. Effect of separation of salted and unsalted serum on bacterial growth at room temperature.

A - serum separated - unsalted

A<sub>1</sub> - serum emulsified - unsalted

B - serum separated - salted

B<sub>1</sub> - serum emulsified - salted.



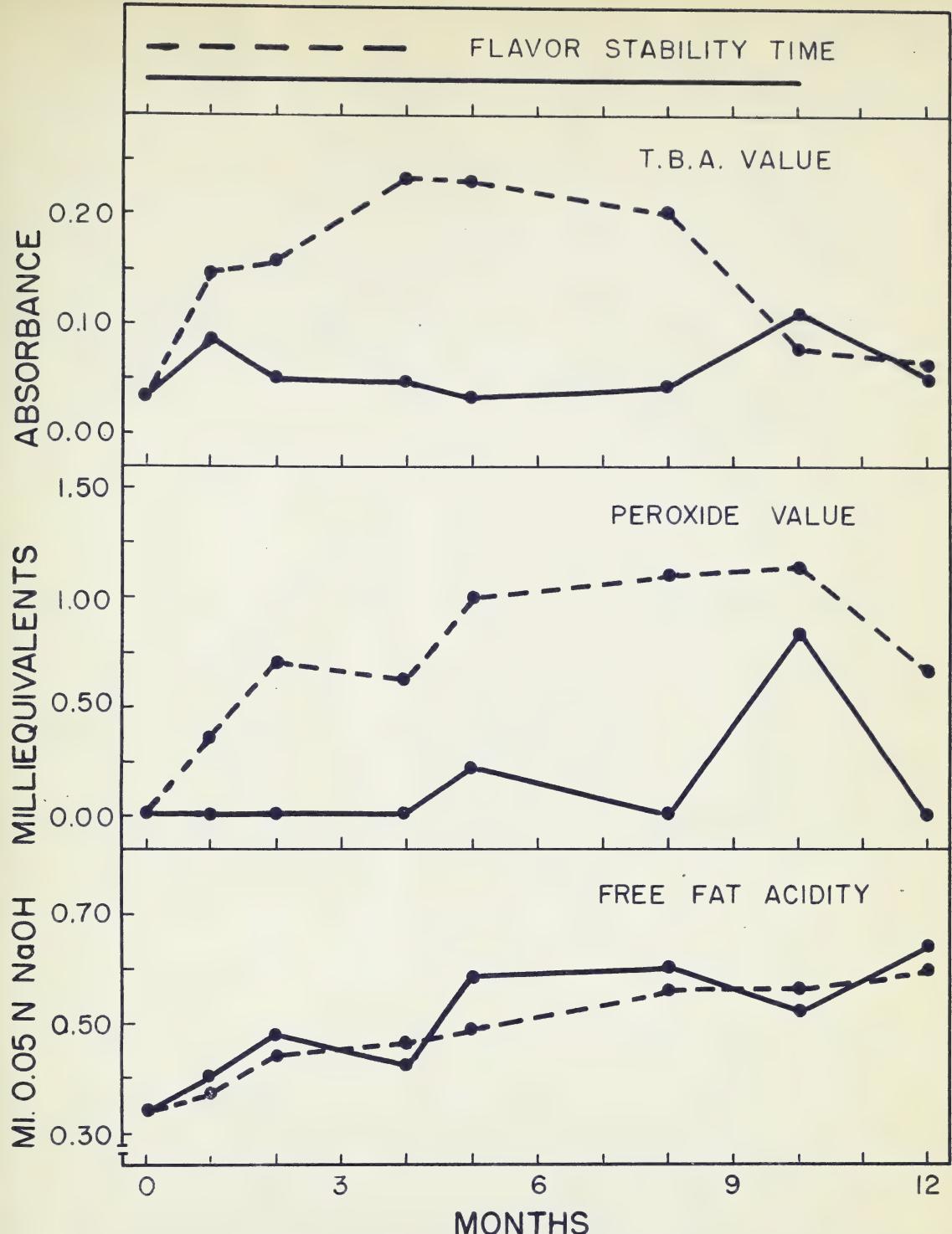


Figure 4. Flavor stability determinations on two butters of the second modification stored at 70°F.

- - - Butter 18 - nitrogen packed containing no additives  
 — Butter 19 - nitrogen packed containing N.D.G.A. and sodium benzoate.



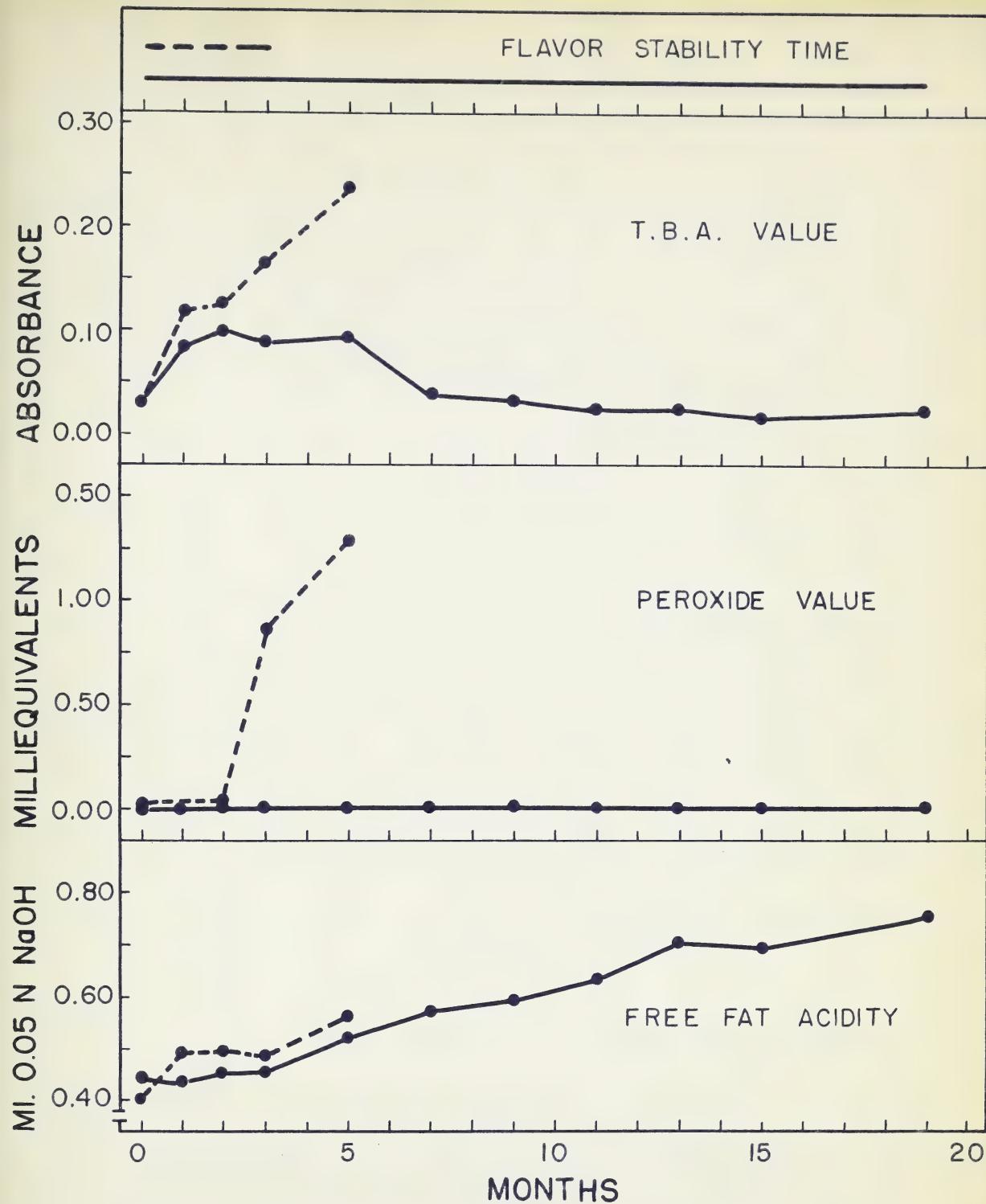


Figure 5. Flavor stability determinations on two butters of the second modification containing no additives stored at 70°F.

— Butter 32 - non-nitrogen packed control  
 — Butter 33 - nitrogen packed.



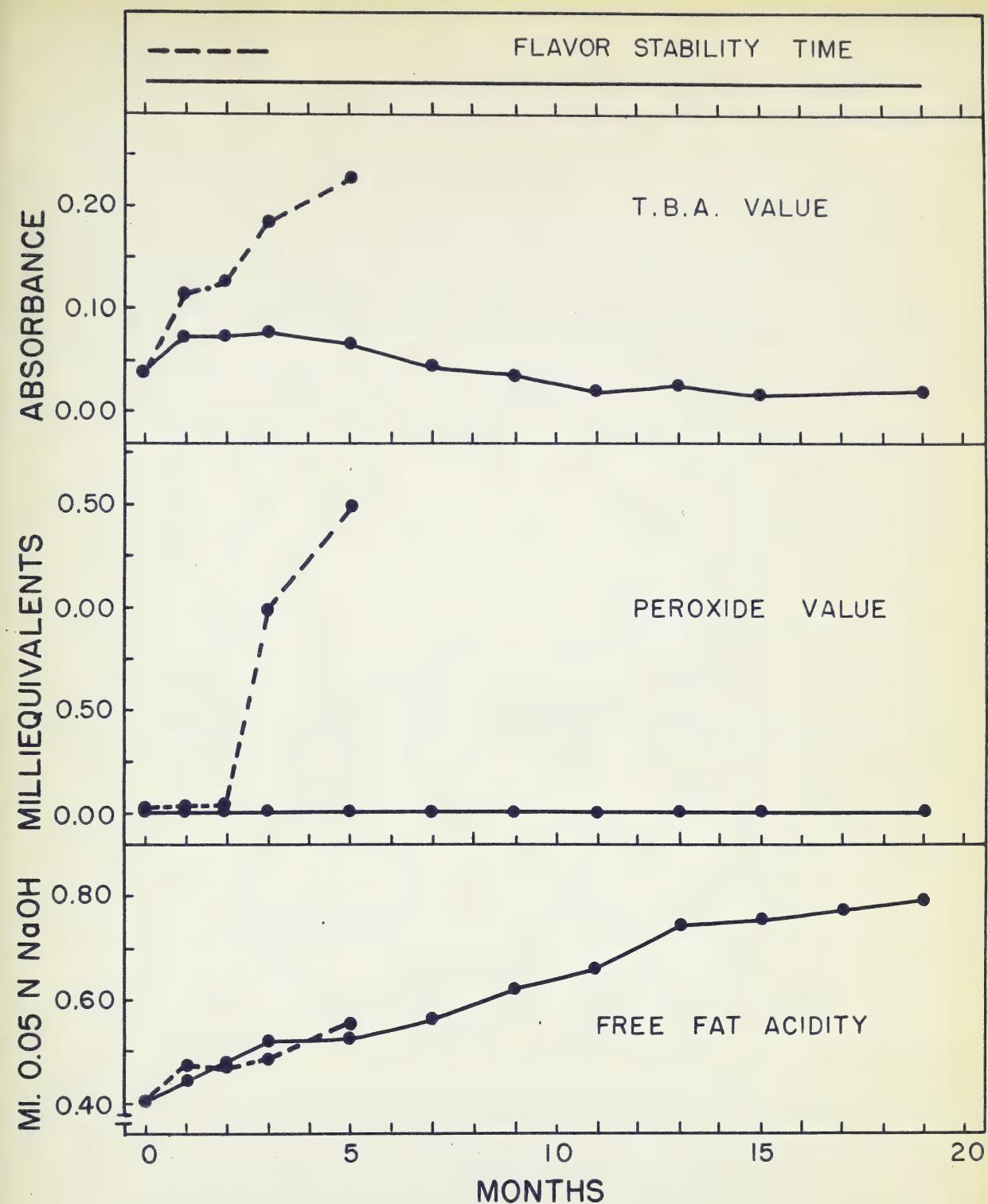


Figure 6. Flavor stability determinations on two butters of the second modification containing Tenox II stored at 70°F.

— Butter 36 - non-nitrogen packed control  
 — Butter 37 - nitrogen packed.



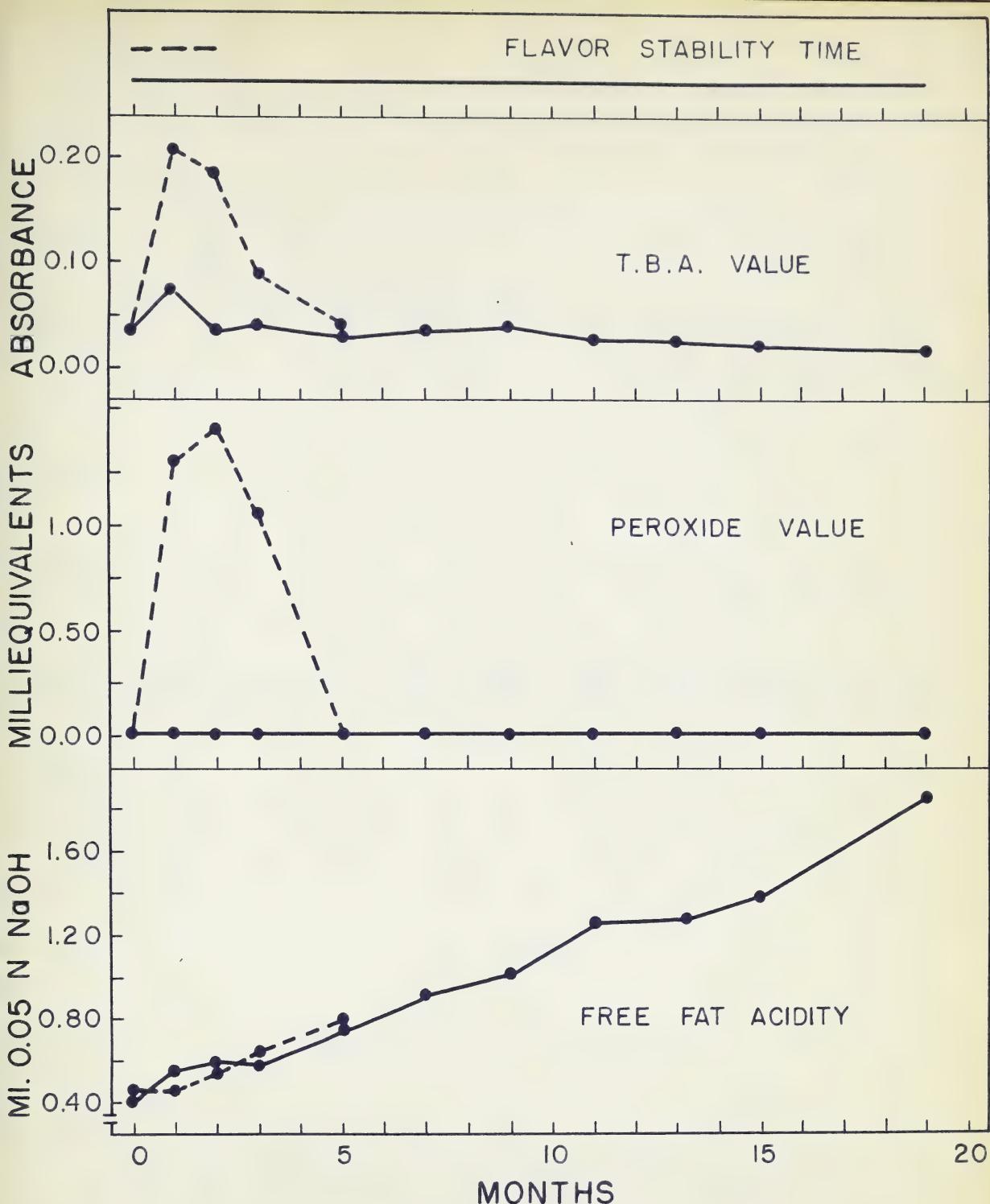


Figure 7. Flavor stability determinations on two butters of the second modification containing no additives stored at 90°F.

— Butter 40 - non-nitrogen packed control  
 — Butter 41 - nitrogen packed.



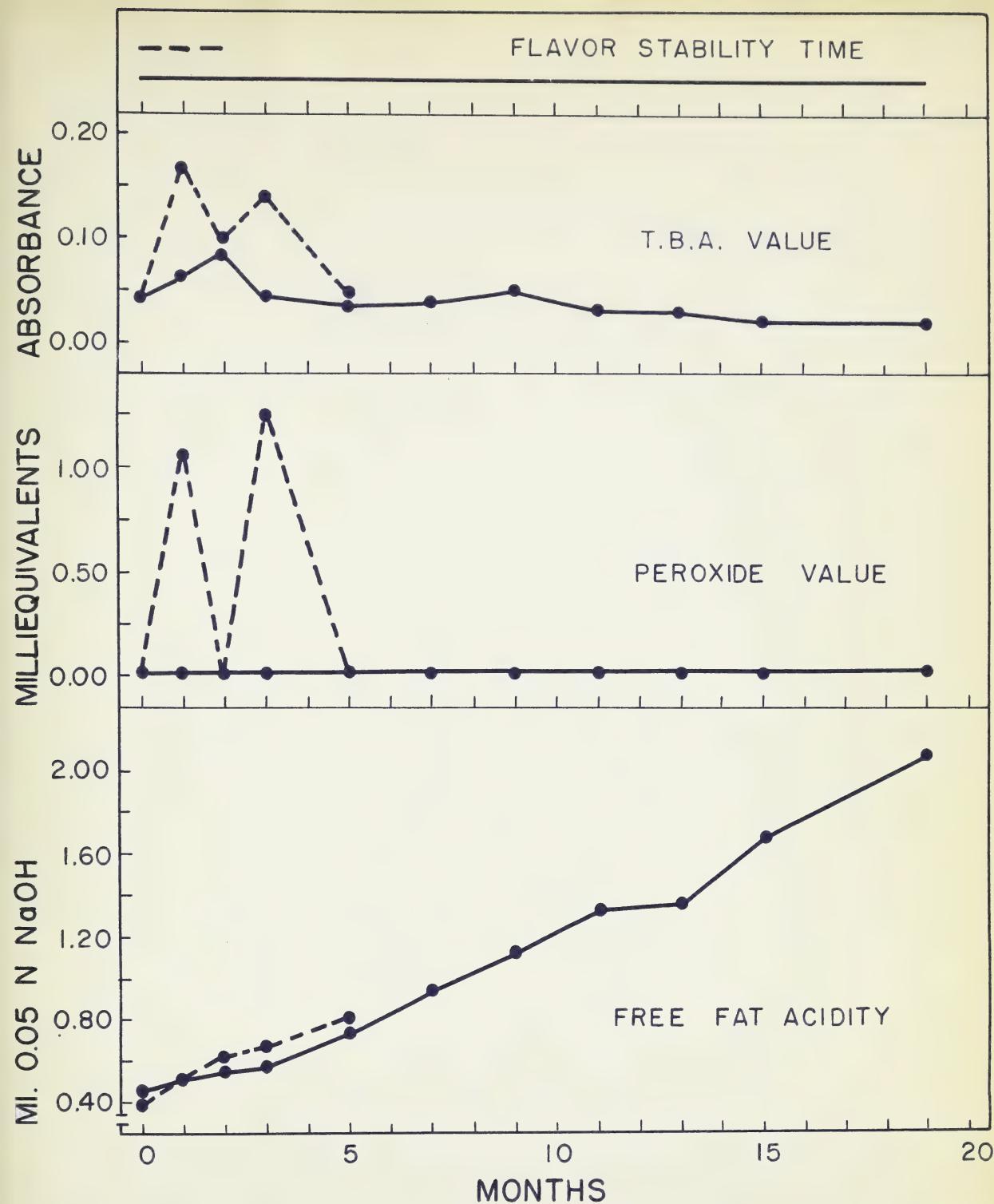


Figure 8. Flavor stability determinations on two butters of the second modification containing Tenox II stored at 90°F.

— Butter 44 - non-nitrogen packed control  
 — Butter 45 - nitrogen packed.









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